Whitman's Pond Vegetation Management Action Plan

Weymouth, Massachusetts

PREPARED FOR:
Town of Weymouth and Whitman's Pond Working Group
75 Middle Street
Weymouth, Massachusetts 02189

PREPARED BY:
ESS Group, Inc.
401 Wampanoag Trail, Suite 400
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ESS Project No. W301-000

September 30, 2013

www.essgroup.com

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Susan M. Kay
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75 Middle Street
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August 27, 2013

Dear Fellow Citizens:

The Whitman's Pond Working Group is pleased to be providing this cover letter for the 2013 Vegetation Management Action Plan for Whitman's Pond. As a critical herring spawning ground, recreational asset, water supply, and central aesthetic feature, Whitman's Pond is an important resource for the town in so many ways. Past efforts to manage vegetation growth have been sporadic and met with partial success. Pond advocates, with varied opinions, recognized the need to initiate a new approach.

In September 2010, Mayor Susan Kay convened a Whitman's Pond "stakeholders" meeting, to bring together the major interests to discuss how the Town could move forward with pond management. This group became the Whitman's Pond Working Group and we have met twelve times since that first meeting. We intend to continue meeting to move the process forward from plan to action.

The Vegetation Management Action Plan was funded by a $24,800 grant from the Community Preservation Committee. The Working Group selected the lake management firm ESS Group, Inc. to prepare the plan in March 2012, after issuing a Request for Responses from qualified lake management consultants. ESS Group conducted field work during the summer of 2012, and in January 2013 provided a draft report for review by the Working Group. The Working Group spent the next several months reviewing the draft and meeting to discuss the preferred management plan. The Working Group held a public meeting on June 10, 2013 to explain the plan and receive comments from the public.

After reviewing the draft plan and the public comments, the Working Group has selected the following elements to form the cornerstone of the Vegetation Management Action Plan:

1. **Hydroraking and Harvesting** – to control nuisance levels of water lilies, as well as for some phragmites control, and to maintain open boating channels and water flow, primarily in the western cove of the Main Basin and in West Cove. The Working

Group is investigating whether it is advisable for the Town to purchase the equipment rather than contract services.

Action item: *Evaluate options and proceed with purchase of equipment or contract of services for 2014 season.*

2. **Winter Drawdown** – to weaken variable milfoil and fanwort through repeated winter drawdowns in the Main Basin and South Cove. Further study is needed to determine if drawdown in West Cove is feasible and cost-effective. The DPW needs to confirm the ability to lower water levels at the Whitman's Pond dam (to be done early Fall 2013). Following this, the Working Group will pursue funding for the required permitting studies.

   Action item: *Request funding for permitting after DPW confirms that the drawdown at the dam is feasible.*

3. **Dredging South Cove** –to increase depths and remove soft sediments in the South Cove. The first priority is to dredge the northern portion of the South Cove. The second priority is to dredge the southern portion of South Cove. Depending on costs and funding availability, these projects would either be done at one time or would be phased.

   Action item: *Obtain funding for permitting.*

4. **Management and Monitoring** – The preferred management alternative consists of multiple techniques, all of which (except for dredging) have to be continually repeated over the years ahead. It is critical that the Town provide a reliable source of funding for both treatment techniques and on-going monitoring. Monitoring will provide us with critical feedback. It will enable us to understand how well the different techniques are working, what their environmental impacts are (both positive and negative), and will guide us in identifying the specific areas that need treatment or management in a given year. Monitoring also enables us to identify the presence of new invasive plants in the pond.

5. **Continuation of the Working Group** – The Working Group members believe the group should continue to meet to oversee the vegetation management program and to address other issues related to Whitman's Pond. The Working Group can periodically hold public meetings to provide information and solicit public feedback on the vegetation management program. The Working Group can also help to coordinate public education and outreach efforts.

6. **Additional Study of the Herbicide Recommendation** – The ESS report recommends partial lake or spot treatments with Sonar (active ingredient fluridone) and Clipper (active ingredient flumioxazin). The Town has used Sonar with some success in West Cove. Clipper was only recently registered for use in aquatic environments in Massachusetts. The Working Group will continue to collect information about these herbicides, and will determine whether, and how, to use them as part of the integrated vegetation management plan for Whitman's Pond.

7. **Stormwater Management** – Although the vegetation management plan did not encompass a review of stormwater management issues, the consultant did highlight two of the most pressing needs. These are: 1) the need for stormwater improvements to reduce sediment loads at the Cynthia Circle outfall into West Cove; and 2) a review of the operation and effectiveness of the Sediment Nutrient Uptake Pond located on the Old Swamp River at Libbey Parkway. The Working Group is in agreement that these are high priority items that need to be addressed.

The Working Group believes that development of this plan has moved the town closer to effective and responsible vegetation management at Whitman's Pond. We hope that you agree and look forward to continued dialogue on protecting and preserving this precious resource.

Sincerely,

The Whitman's Pond Working Group

Mayor Susan M. Kay
Town Council President Artie Mathews
Jeffrey Bina , Public Works Director (left town service July 2013)
Andrew P. (Chip) Fontaine, Town Engineer
Jim Clarke, Director, Planning & Community Development
Mary Ellen Schloss, Conservation Administrator
George Loring , Herring Warden and Conservation Commissioner
Scott Dowd, Conservation Commissioner
Phil Lofgren, Assistant Herring Warden
Trish Pries, President, Whitman's Pond Association
Tom Daru, Whitman's Pond Association

WHITMAN'S POND VEGETATION MANAGEMENT ACTION PLAN
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1.0 INTRODUCTION

ESS Group, Inc. (ESS) has prepared this Vegetation Management Action Plan for Whitman's Pond on behalf of the Town of Weymouth (Town) and the Whitman's Pond Working Group. The objective of this plan is to provide the Town with a framework for managing nuisance vegetation at the pond while preserving or improving aquatic habitat, water supply, recreational opportunities, and overall water quality.

This Vegetation Management Action Plan provides background information on existing conditions within Whitman's Pond and its watershed and offers cost-effective and environmentally sound recommendations for the pond's future management.

1.1 Whitman's Pond Description

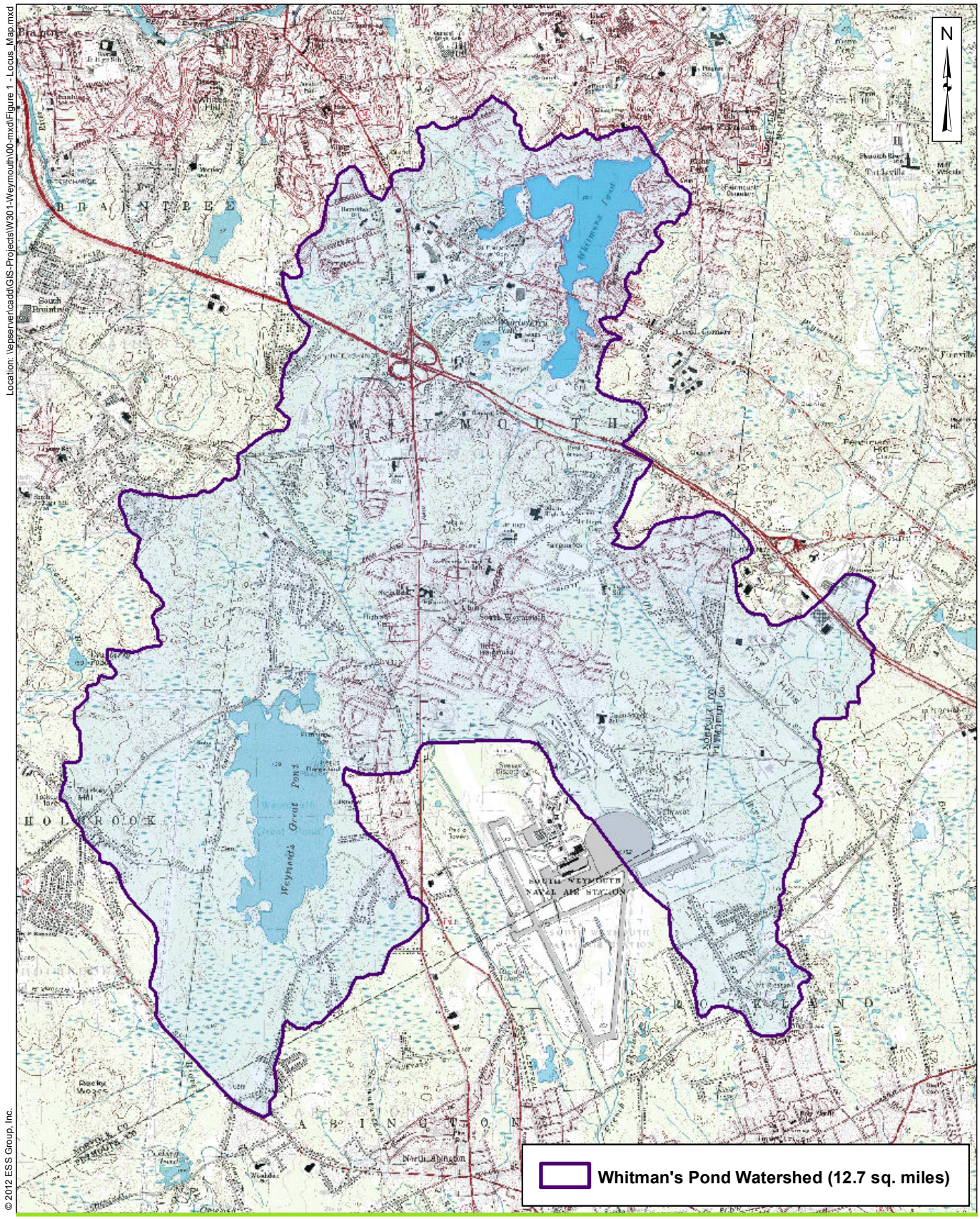
Whitman's Pond is an approximately 200-acre waterbody that is divided into three basins, including the Main Basin, the West Cove, and the South Cove. The South Cove is used to supplement the Town's water supply via a pump station on Washington Street. The West Cove is former swampland that is connected to the Main Basin by a culvert during high water but is isolated during drought periods. The shorelines of all three basins are primarily private residential. Public access boat launches are located on the West Cove and Main Basin. However, additional public shoreline areas are used as unimproved access for fishing, boating, ice skating, wildlife observation, and other types of recreation.

The growth of exotic invasive weeds such as fanwort (*Cabomba caroliniana*) and variable-leaf milfoil (*Myriophyllum heterophyllum*) plagues Whitman's Pond at nuisance levels and poses the main management challenge. Fanwort, in particular, has been a nuisance for over three decades and is now the dominant aquatic plant in the pond. Additionally, the exotic invasive curly-leaf pondweed (*Potamogeton crispus*) has been documented in smaller beds.

The Whitman's Pond watershed spans approximately 13 square miles and is located mostly within Weymouth but also includes portions of the towns of Braintree, Holbrook, Hingham, Abington, and Rockland as well as the former South Weymouth Naval Air Station (Figure 1). Two primary tributaries drain the Whitman's Pond watershed. Old Swamp River is the largest tributary and drains the southeastern portion of the overall watershed into the South Cove of Whitman's Pond. This includes contributing areas of Rockland, Hingham and the South Weymouth Naval Air Station property. The second primary tributary is the Mill River, which enters the Main Basin from the west and drains the southwestern portion of the overall watershed. This includes contributing areas of Abington, Holbrook, and Braintree as well as outflow from Weymouth Great Pond and urban runoff from some of the more densely developed industrial and commercial districts of Weymouth. Several smaller tributaries also enter the pond, mainly through the West Cove. Additionally, as many as 50 stormwater outfalls dot the shoreline of Whitman's Pond and directly contribute flow to the pond during wet weather (Metcalf and Eddy 1983, BETA Group 2001 and 2004).

Water flow between the West Cove and Main Basin is controlled by a single box culvert under Middle Street. Flow through the culvert is obstructed by boards but is able to pass through leaks in the boards (Brad Chase, Massachusetts Division of Marine Fisheries, personal comm.). Flow between the South Cove and Main Basin can be manipulated by way of a sluice gate and stoplogs located at the Washington Street bridge.

Water from the Main Basin of Whitman's Pond leaves via the outlet at the northeast extreme of the pond (Figure 2). Here, flow leaves through the main spillway as well as a Denil fishway. A portion of high flows is diverted through a series of four diversion outlets. Additional manipulation of water level control is available through various other measures, including a gate valve and flashboards. Outlet flow briefly enters Iron Hill Pond before continuing through a series of five additional fishways on Herring Brook, the Weymouth Back River, and eventually out to Hingham Bay.



VEGETATION ACTION MANAGEMENT PLAN
Whitman's Pond, Weymouth, MA

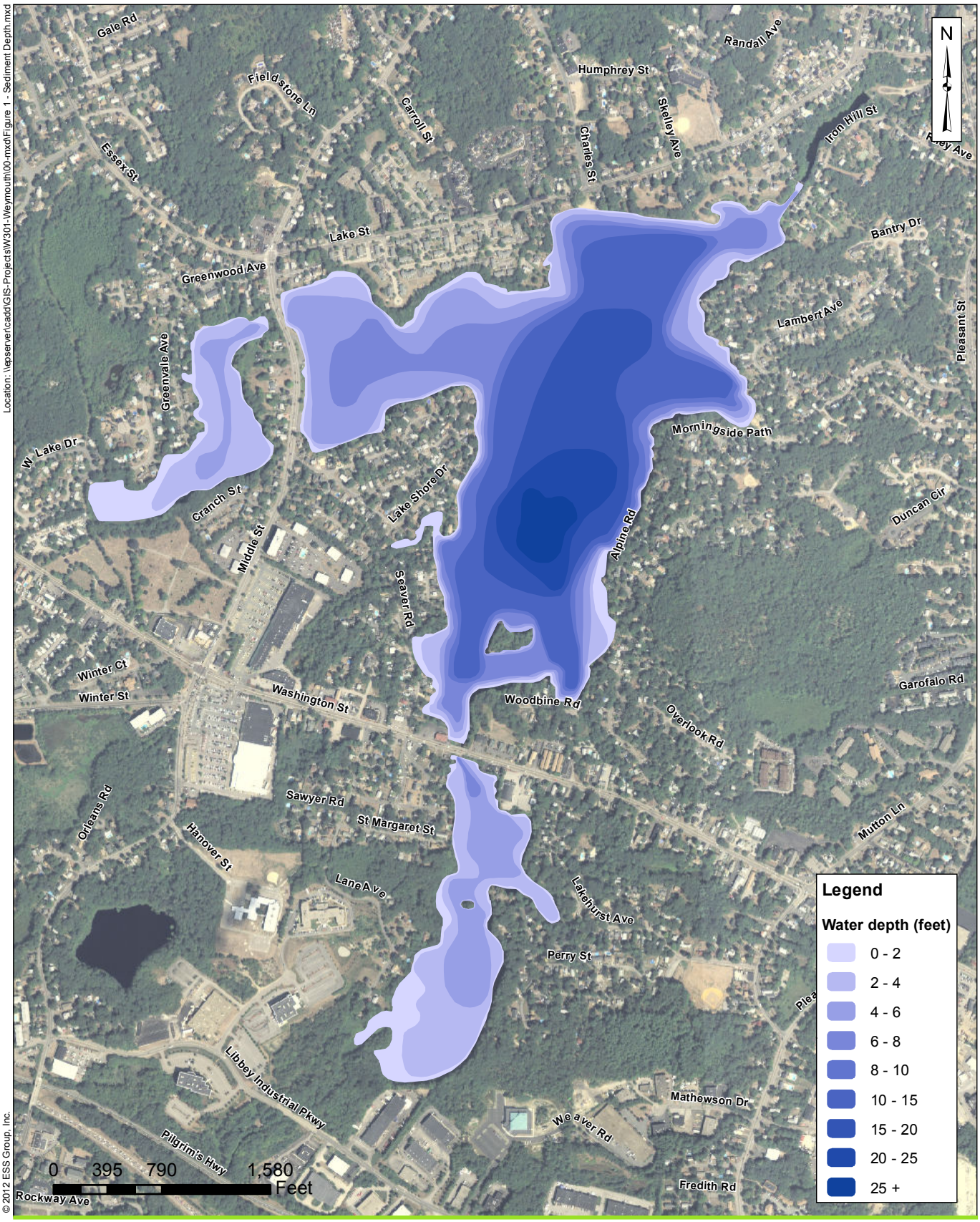
Locus Map

1 in = 4,000 ft

0 0.25 0.5 Miles

Source: 1) USGS 1:24,000 topographic basemap
2) ESS Watershed boundary data

Figure 1



Vegetation Management Action Plan
Whitman's Pond, Weymouth, MA

1 in = 1,000 ft

Source: 1) NAIP Orthophotography, 2009
2) ESS Bathymetry Data

Bathymetry

Figure 2

Outlet structures at Whitman's Pond include the fishway (left), main spillway (center), and flow diversion gates (right).

The Weymouth Back River Area of Critical Environmental Concern (ACEC) begins at the Whitman's Pond dam and continues along downstream portions of Herring Brook, the Weymouth Back River, and bordering lands. A series of six fish passage structures allow alewife (*Alosa pseudoharengus*) and other diadromous fishes to bypass dams in the Weymouth Back River ACEC and enter Whitman's Pond for spawning. The waters of Whitman's Pond and Herring Brook support thousands of spawning anadromous alewife, which constitutes one of the largest river herring runs north of Cape Cod (Back River Committee undated). In light of these facts, vegetation management efforts in Whitman's Pond will need to be cognizant of and preserve or improve these ecological services.

Sediment and excess nutrients are transported to the pond from its tributaries as well as from the numerous stormwater outfalls that discharge directly to the pond around its perimeter. The sediment accumulation, excess nutrients in the water column and dense growths of exotic aquatic plants contribute to a somewhat degraded condition, particularly in shallower parts of the Main Basin and West Cove. Weed growth and water and sediment quality concerns reduce the pond's ability to provide more desirable aquatic habitat conditions and limit the full potential for recreational opportunities. This Vegetation Management Action Plan examines Whitman's Pond in further detail and provides management recommendations to restore recreational opportunities and improve habitat and water quality without negatively impacting the ecology of the system or the critical resources that the system supports.

2.0 METHODS AND APPROACH

The field studies and data evaluation supporting our analysis of the Whitman's Pond system were conducted from April through September 2012 and included a review of existing data and reports, GIS mapping, field data collection, and data analysis. The methods and approach specific to each task are described in the following sections.

2.1 QAPP Development

ESS developed a Quality Assurance Project Plan (QAPP) for the work completed under this project (Appendix A). The QAPP serves as a project-specific guidance document to ensure that data quality objectives are met during field and laboratory data collection and analysis. A primary advantage of working under an approved QAPP is that the data collected are well-suited to support state and federal grant program applications for future pond study or management work.

This project's QAPP included plans for the data collection, analysis, and quality control protocols covering all data generating aspects of the project. ESS submitted a draft QAPP to the Whitman's Pond Working Group for review and comment. Comments received from the Whitman's Pond Working Group were incorporated into the QAPP and it was submitted to US Environmental Protection Agency (US EPA) Region 1 for review on July 6, 2012. US EPA approved the QAPP pending attention to two minor

comments on July 30, 2012, which were addressed by ESS. A final QAPP incorporating the requested changes was sent to US EPA on August 9, 2012. Notification of US EPA's QAPP review was also sent to the Massachusetts Department of Environmental Protection (MassDEP) on July 30, 2012.

2.2 Review of Previous Studies

The Whitman's Pond Working Group provided ESS with a number of existing reports on Whitman's Pond at the project kick-off meeting held on April 2, 2012. The primary project reports and studies that ESS reviewed are summarized in Table A.

Table A. Summary of Previous Studies and Reports Reviewed by ESS

Report/Study	Year	Author	Brief Description
Whitman's Pond Pre-treatment Survey Results and Management Plan	2010	Aquatic Control Technology, Inc.	Aquatic plant mapping and plan for pond-wide herbicide treatment in six zones
Quality Assurance Program Plan for Water Quality Measurements Conducted for Diadromous Fish Habitat Monitoring (Technical Report TR-42)	2010	Massachusetts Division of Marine Fisheries	Describes the protocols used by the Division of Marine Fisheries for alewife habitat monitoring in Whitman's Pond.
Whitman's Pond Comprehensive Investigation, Evaluation, and Hydrological Study	2004	BETA Group, Inc.	Summarized previous studies and provided new sediment and storm water data. Recommended in-pond and watershed stormwater management actions.
Habitat Study of Whitman's Pond	2001	BETA Group, Inc.	Summarized previous studies and provided water quality, sediment, and biological data. Recommended additional stormwater and septic studies.
Annual Report for Whitman's Pond Project	1998	Ambient Engineering, Inc. & Ocean Arks International	Evaluated existing pollution control measures and made recommendations for continued pond management. Recommendations focused on stormwater management, SNUP management and optimization, and the "Lake Restorer" biological filtration and incubation system in the West Cove.
Whitman's Pond Management Plan	1998	Whitman's Pond Restoration Committee and Whitman's Pond Association	Summarized findings of previous studies. Also reported on historical and current events around the Pond and in the watershed. Proposed management goals, notably invasive weed control, pond dredging (to improve water supply and pond health), and Canada Goose control.

Table A. Summary of Previous Studies and Reports Reviewed by ESS

Report/Study	Year	Author	Brief Description
Diagnostic Feasibility Study Western Basin of Whitman's Pond	1997	Lycott Environmental, Inc.	Mapped bathymetry, estimated sediment volume, and collected water quality data in the West Cove. Also provided limited sediment quality data. Treated West Cove with Fluridone to control fanwort. Primarily recommended herbicide control of nuisance plants in the West Cove and improvements to stormwater and septic management in the watershed.
Inventory of Natural Resources and Land Use in the Weymouth Back River ACEC	1997	Jennifer Myers (for Back River Committee)	Provided an inventory of natural features, human uses, and regulations pertaining to the ACEC.
Water Supply Assessment for Mill River and Old Swamp River Basins	1989	CDM	Investigated safe yield of Town surface and groundwater sources and made recommendations for preserving water supply quality and quantity.
Feasibility Study of Lake Restoration	1983	Metcalf & Eddy, Inc.	Presented analyses of pond and watershed hydrology, nutrient, bacteria and sediment loading, and biological inventories. Suggested restoration recommendations including clean up of the pond shorelines, reduction of in-pond phosphorus concentrations, elimination of nuisance plants, reduction of aquatic toxicity, provision of constant downstream flow release, and reduction of water intake clogging. Also included the recommendation to construct a sedimentation/ nutrient uptake pond (SNUP) on the Old Swamp River.
Whitman's Pond Diagnostic Study	1983	Massachusetts Department of Environmental Quality – Division of Water Pollution Control	Extensive investigation of water quality, sediment quality, hydrology, recreational use, and biology of Whitman's Pond. Recommendations in Metcalf and Eddy (1983).
Ecology of the Back River (Draft)	Undated (Provided to ESS in 2012)	Back River Committee	Broad summary of historical, socioeconomic, and ecological conditions in the region and watershed.

In addition to these reports and previous studies, the Town also furnished the following sources of information to assist with development of the Vegetation Management Action Plan.

- Washington Street Pumping Station monthly raw water pumping volumes for the period from 2004 to 2011.
- Completed alewife habitat assessment data sheets. The preliminary data contained in these sheets were generated by Brad Chase of the Massachusetts Division of Marine Fisheries over the May to September period of 2011.

ESS compiled additional information on current watershed and pond features from Massachusetts Geographic Information System (MassGIS) data. We also compiled supplemental information from the following sources:

- Massachusetts Year 2010 Integrated List of Waters. Prepared by the Massachusetts Department of Environmental Protection, Division of Watershed Management.
- Historical fish survey reports maintained by the Massachusetts Division of Fisheries and Wildlife. The files reviewed include surveys from 1905, 1956, 1957, 1958, and 1983.

2.3 Bathymetry and Isopach Survey

A bathymetric (water depth) and sediment isopach (unconsolidated sediment thickness) survey was completed at Whitman's Pond on June 7, 2012. The purpose of the survey was to collect data for use in assessing the feasibility of pond management options, particularly dredging and drawdown. Prior to conducting the survey, 143 bathymetry points and 92 sediment isopach points were laid out in each basin using a modified point-intercept method, as outlined in the project QAPP (Appendix A). The planned sampling points were uploaded onto a Trimble GeoXT Global Positioning System (GPS) and used in the field to navigate to each sampling station during the survey. Based on field conditions, some sampling points were shifted, while others were added or removed. The actual number of points surveyed for bathymetry and sediment isopach were 162 and 117, respectively.

Vertical control points were identified for each basin on the day of survey. In the main basin, the elevation at the spillway staff gage was recorded. In the South Cove, the elevation of the water surface in relation to the Washington Street flow gate structure was measured. In the West Cove, the elevation of the water surface above the box culvert invert was measured.

An extendible carbon steel tile probe was used to measure water depth and total depth. Total depth was obtained by pushing the tile probe into soft sediments until first refusal at a harder underlying substrate was reached. Water depth and total depth data were recorded in a GPS data dictionary. Soft sediment thickness was calculated as the difference between total depth and water depth. Positions were downloaded, differentially corrected and exported to GIS to create bathymetry and sediment isopach figures.

2.4 Sediment Sampling

Collection of sediment samples is used to examine physical and chemical sediment quality. Samples document physical characteristics and identify levels of potential contaminants that could pose challenges for pond management techniques that involve bottom disturbance (e.g., dredging). The sediment sampling conducted at Whitman's Pond was designed as a screening process to identify the nature and severity of sediment contamination (if present) in each basin of the pond, as well as the Mill River.

The initial round of sediment sampling at Whitman's Pond included the Main Basin, Mill River and South Cove and was completed on July 19, 2012. A second round of sampling was conducted on August 29, 2012 in the West Cove. Sediment sampling locations were developed in consultation with the Whitman's

Pond Working Group. Prior to collecting sediment, the coordinates of 18 targeted sediment core locations were uploaded to a sub-meter accurate Trimble GeoXT GPS for field navigation. The sediment core locations were selected to characterize areas of Whitman's Pond under consideration for dredging.

Sample sediment cores were recovered from the pond bottom using an extendible Russian peat corer at locations shown in Figure 3. ESS photographed each sediment core (Appendix B) and characterized the core color and texture.

The 18 individual sediment cores were composited into a total of six sediment samples (MB, MR, SC1, SC2, SC3, and WC) and submitted to the laboratory for analysis. Each sample consisted of three individual sediment cores (SC1-1, SC1-2, SC1-3, etc.). Compositing was accomplished by homogenizing each set of three cores with a stainless steel spoon in a stainless steel bowl prior to removing sample material for laboratory analysis. An exception to this protocol was made for volatile organic compounds (VOCs), which were sampled from individual cores prior to compositing, in order to avoid sample loss through volatilization.

Bulk physical and chemical analysis was conducted on the six composite samples. Sediment samples were analyzed for the following parameters: arsenic, cadmium, chromium, copper, lead, mercury, nickel, zinc, polychlorinated biphenyls (PCBs), volatile organic compounds (VOCs), polynuclear aromatic hydrocarbons (PAHs), extractable petroleum hydrocarbons (EPH's), pesticides, total organic carbon (TOC), moisture content, and ASTM grain size analysis.

2.5 Water Quality Sampling

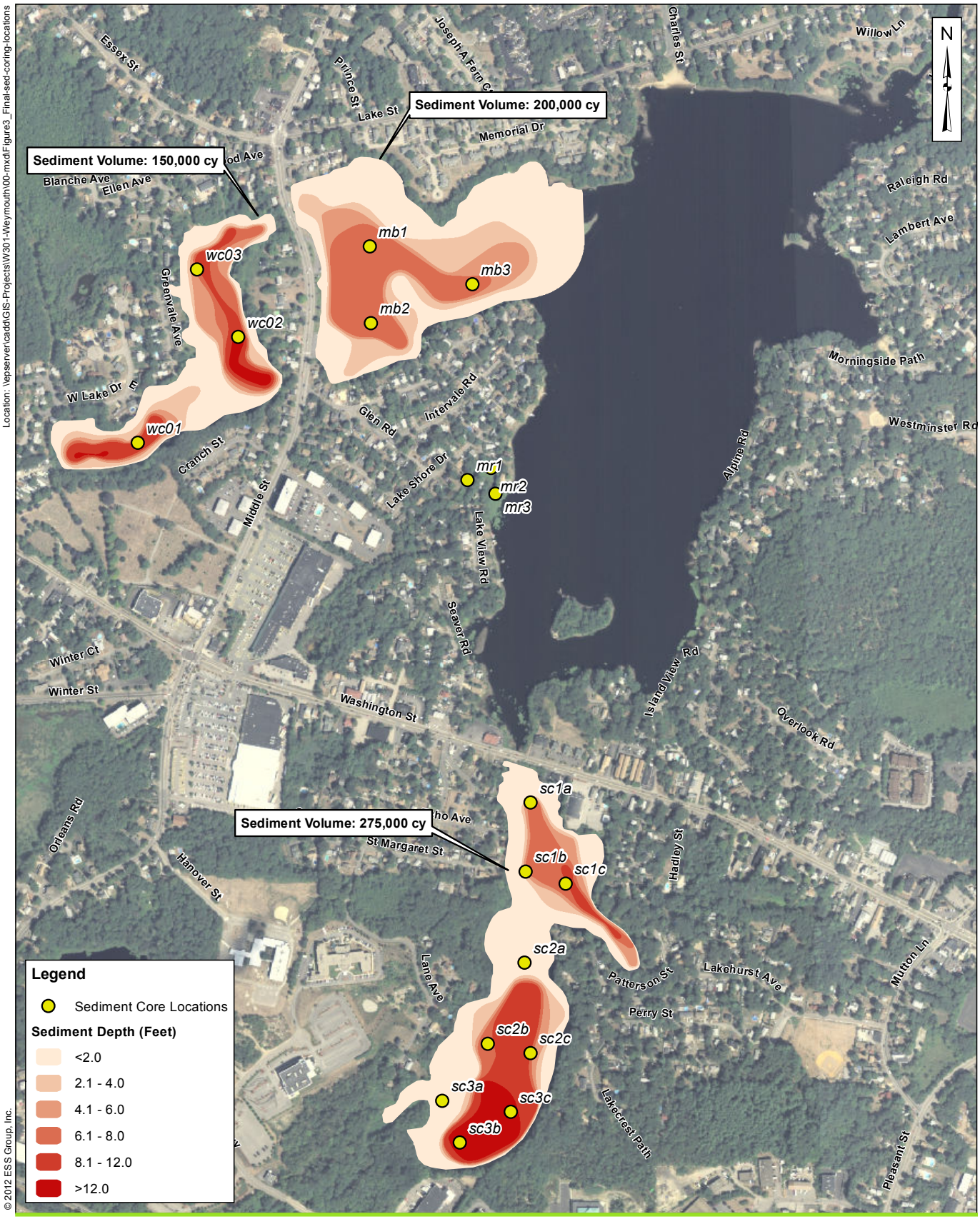
ESS collected a single round of water quality data to help establish baseline conditions in the pond during the late summer period. Data were collected at the pond surface and bottom (MB-S and MB-B, respectively), at one location in the West Cove (WB) and in the South Cove at the mouth of the Old Swamp River (SB) for a total of four sampling locations (Figure 4). ESS also measured in-pond water clarity (Secchi depth) and collected temperature and dissolved oxygen data through the water column at the deepest location in the pond. These data were used to develop a full vertical profile of Whitman's Pond and estimate the areal extent of hypoxia (low oxygen conditions) in the pond.

Field parameters measured at all locations by ESS included pH, dissolved oxygen, temperature, specific conductance, turbidity, and color. In addition to the field parameters, ESS collected samples for laboratory analysis of total phosphorus and total nitrogen by a state-certified laboratory (Premier Laboratory of Dayville, Connecticut). Water quality sampling and analysis was completed in accordance with the project QAPP (Appendix A).

As a QA/QC measure for the water quality sampling activities, one duplicate sample was sent to the laboratory.

2.6 Fish and Wildlife

The objective of the fish and wildlife assessment for Whitman's Pond was not to create a complete seasonal inventory but rather to characterize the pond's fish and wildlife communities and hydraulically connected wetlands. Qualitative observations were made during field visits spread out over several months. Particular attention was given to potential nuisance species (e.g., Canada Goose) as well as those that may be sensitive to certain management options (e.g., drawdown).



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VEGETATION MANAGEMENT ACTION PLAN
Whitman's Pond, Weymouth, MA

1 in = 800 ft

0 500 1,000 Feet

Source: 1) NAIP Orthophotography, 2009
2) ESS Sediment Data

Sediment Isopach and Core Locations

Figure 3

Fish Habitat Suitability Assessment

It was originally anticipated that the primary source of habitat suitability information for alewife at Whitman's Pond would be the study currently underway through DMF, which was being conducted under a separate QAPP (Chase 2010). Although the field portion of the study was completed in 2012, as of the time of writing this report, it was not certain when the final results of the DMF study would be available. However, ESS did contact Brad Chase, the primary investigator on the DMF study, in November 2012 to discuss preliminary results.

Sediment (substrate), plant cover, and water depth information collected as part of this study were used to develop maps of suitable habitat for fish species known to inhabit Whitman's Pond. Habitat suitability assessments were based primarily on the USFWS Habitat Suitability Indices published for individual species (where available) but were also supplemented with other readily available habitat suitability information sources and best professional judgment.

Fish and Wildlife Observations

Fish and wildlife use of Whitman's Pond and adjacent habitats was observed at various times over the course of this study. Qualitative observations were made during field visits that occurred on April 2, June 7, July 19, August 29, and September 24, 2012. Groups of species observed included birds, mammals, fish, reptiles, amphibians, and macroinvertebrates.

On September 24, 2012, targeted qualitative observations of fish and macroinvertebrates were made in Whitman's Pond. Fish were observed using a Marcum vs625 color underwater camera and minnow traps. Aquatic macroinvertebrates were sampled and observed using a D-frame kick net and clam rake in accordance with the methodology described in the QAPP (Appendix A). Macroinvertebrate observations were focused in four key areas of the pond based on their likelihood to support freshwater mussel populations (Figure 4).

Wetland Characterization

Hydraulically connected wetlands around Whitman's Pond were identified and characterized by a Professional Wetland Scientist (PWS) on August 29, 2012 and September 24, 2012. The level of characterization was sufficient for impact assessment of potential pond vegetation management options.

2.7 In-lake Vegetation Assessment

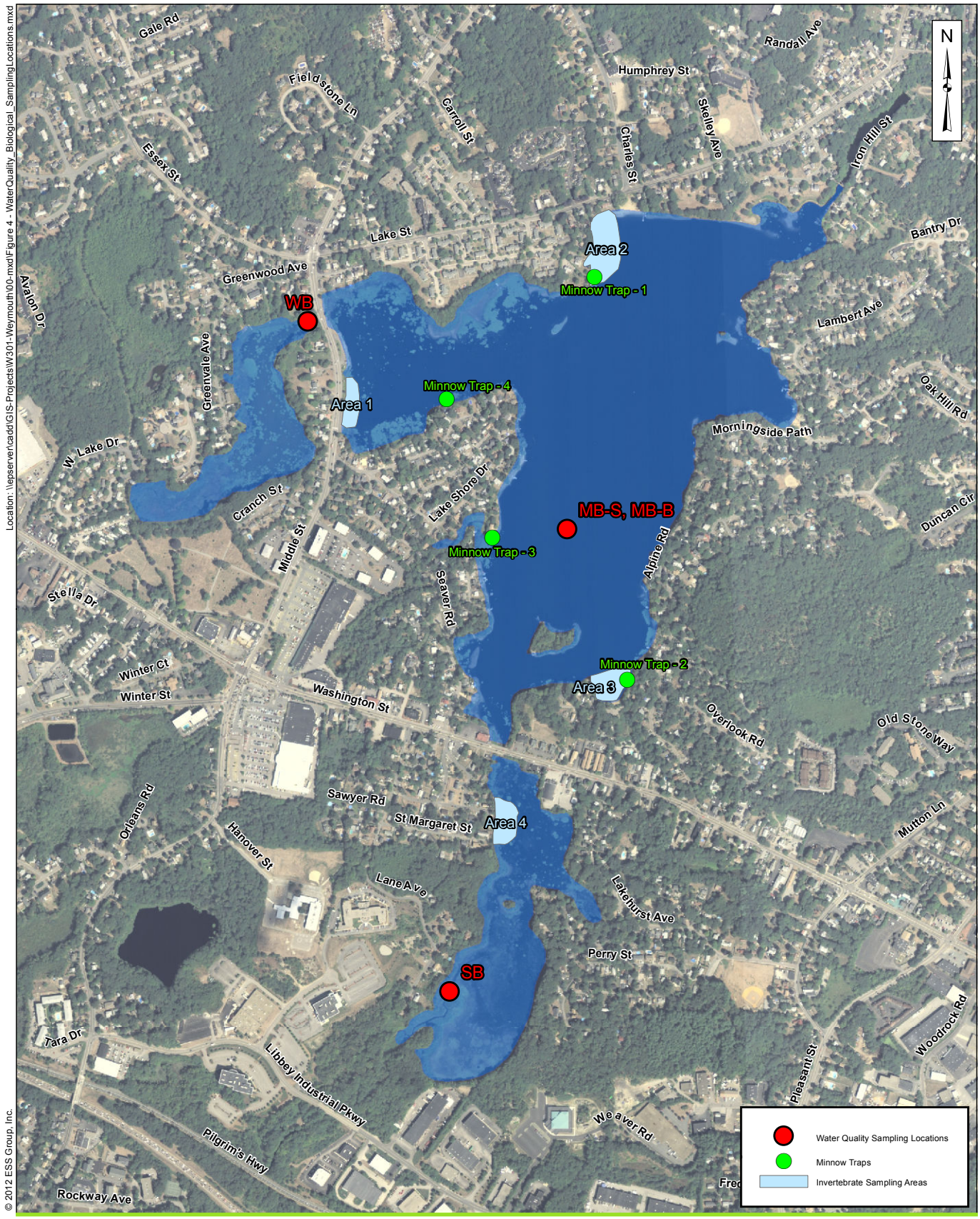
ESS mapped the aquatic plant community in Whitman's Pond on August 29 and September 24, 2012 to update current species composition, cover, and biovolume in each basin from previously mapped conditions.

Aquatic plants were mapped in accordance with the QAPP (Appendix A), although fewer (109) point locations were surveyed than planned. Mapping equipment included a Marcum vs625 color underwater camera and plant rakes. Dominant species, percent cover, and biovolume were recorded at each point and positions collected with a Trimble GeoXT GPS capable of sub-meter accuracy.

Plant species encountered were identified in the field by qualified staff. Taxonomic keys (including Crow and Hellquist 1982, New England Aquarium 1999, Crow and Hellquist 2000) were consulted as needed to assist in aquatic plant identification.

2.8 Recreational Use Assessment

Recreational uses of Whitman's Pond were assessed by reviewing existing data, visiting public access locations, and gathering information about the current activities at the pond from members of the Whitman's Pond Working Group. These uses were factored into the feasibility assessment and recommendations.



VEGETATION ACTION MANAGEMENT PLAN
Whitman's Pond, Weymouth, MA

1 in = 1,000 ft

0 500 1,000 Feet

Source: 1) MassGIS, Orthos, 2009

Water Quality and Biological Sampling Locations

Figure 4

2.9 Hydrologic/Hydraulic Assessment

A hydrologic budget models water inflow, storage capacity and water outflow from the pond based on the hydrologic cycle. Sources of water inflow include precipitation onto the pond surface as well as the associated overland runoff, direct stream flow from tributaries, and groundwater seepage along the margins of the pond. Evapotranspiration, groundwater recharge and direct outflow via the pond outlet all lead to losses of water from the pond. The difference between the sum of the inflows and sum of the outflows determines the storage volume of the pond at a given point in time.

To complete the hydrologic assessment for Whitman's Pond, ESS reviewed existing hydrologic data, including previous reports and USGS gaging station discharge records for the Old Swamp River at Route 3 (USGS ID 01105600), the fish ladder at Iron Hill Street (USGS ID 01105608), and the Whitman's Pond dam/flood diversion outlet (USGS ID 01105606). Streamflow inputs from the two primary tributaries to the pond (Old Swamp River and Mill River) and total surface outputs were further estimated using the USGS StreamStats online application for Massachusetts (USGS 2012). Data on precipitation and evapotranspiration were based on long-term averages from the Logan Airport weather station in Boston (NRCC 2012). An estimate of the rate of groundwater movement into the pond was based on averages obtained for southern New England ponds of similar morphometry. These data were examined in the context of the updated bathymetry data to assess the feasibility of drawdown as a management action in Whitman's Pond.

2.10 Water Supply Resource Assessment

ESS reviewed Town-provided records of water withdrawal at the Washington Street pump station for the 2004 to 2011 period. Patterns in water withdrawal were factored into the feasibility assessment of management actions at Whitman's Pond.

3.0 RESULTS

The results of each component of the study are presented in the following sections. Results include data acquired from previous studies, field collection, desktop review and limnology modeling.

3.1 Quality Assurance/Quality Control

ESS received two minor comments from the EPA Region 1 reviewer. These comments were addressed and incorporated into the final QAPP document (Appendix A). The project complied with the final QAPP, with the exception of the following deviation.

- A QA/QC completeness goal of 80% was outlined in the QAPP for bathymetry and isopach mapping, aquatic plant mapping, and water quality and sediment sampling. This goal was met for each of these items except aquatic plant mapping, which was 76% complete. The reduction in points mapped was related to the fact that plant growth was both excessive and homogeneous over large portions of the South Cove and West Cove while being absent over much of the Main Basin. Also, during the course of the field work, it was determined that no significant plant growth occurred below a depth of 11 feet. Therefore, a number of the originally planned points in deeper waters were eliminated from the Main Basin. However, areal coverage of the mapped locations extended across all key areas of each three basins of the pond. Additionally, ESS also used field sketches to better delineate the actual extent of aquatic plant beds beyond simple point data. Therefore, the slightly lower than targeted number of plant mapping points did not likely reduce the quality of the in-pond vegetation maps produced as part of this project.

A QA/QC precision goal was outlined in the QAPP for water quality sampling. Based on analysis of field duplicates, the Relative Percent Difference (RPD) for total phosphorus was zero and the RPD for total nitrogen was 12.4, both of which are within the goal of 20 for RPD.

All other sampling protocols and goals were met without deviations from the QAPP.

3.2 Bathymetry, Hydrology, and Water Supply Assessments

Results of water depth surveys were used to create a bathymetric map for the pond (Figure 2). The West and South Coves of Whitman's Pond are both shallow (average depth less than 4.0 feet deep), while the Main Basin has an average depth of 10.2 feet. The deepest point in Whitman's Pond is in the south central portion of the Main Basin, where the depth is 26.5 feet. The total volume of water in the Main Basin of the pond is estimated to be approximately 489.5 million gallons with about 31 million gallons in the South Cove and an additional 14 million gallons in the West Cove (Table B).

The dam forming the next impoundment downstream from Whitman's Pond (Iron Hill Pond) was rehabilitated in 2012 and should be structurally sound to pass flows from the outlet (spillway and fishway) at Whitman's Pond dam.

Based on previous studies, available climate data, and basic hydrologic modeling, the average flow through Whitman's Pond is estimated to be approximately 21.88 cfs (Appendix C). The inputs contributing to this total can be broken into direct precipitation, groundwater, and surface water. Annual direct precipitation into Whitman's Pond averages 42.53 inches, which is equivalent to a flow of 0.61 cfs (approximately 3% of total input) over the course of the year when evapotranspiration is subtracted. As indicated in previous studies, groundwater is likely to contribute only a minor portion of the hydrologic input to the pond, approximately 0.01 cfs (less than 1% of total input). Primary surface hydrologic inputs are from the two tributaries, Mill River and Old Swamp River with additional flow from minor unnamed tributaries, stormwater inputs, and overland runoff. Surface inputs account for the bulk of hydrologic inputs to Whitman's Pond (21.3 cfs or 97% of the total).

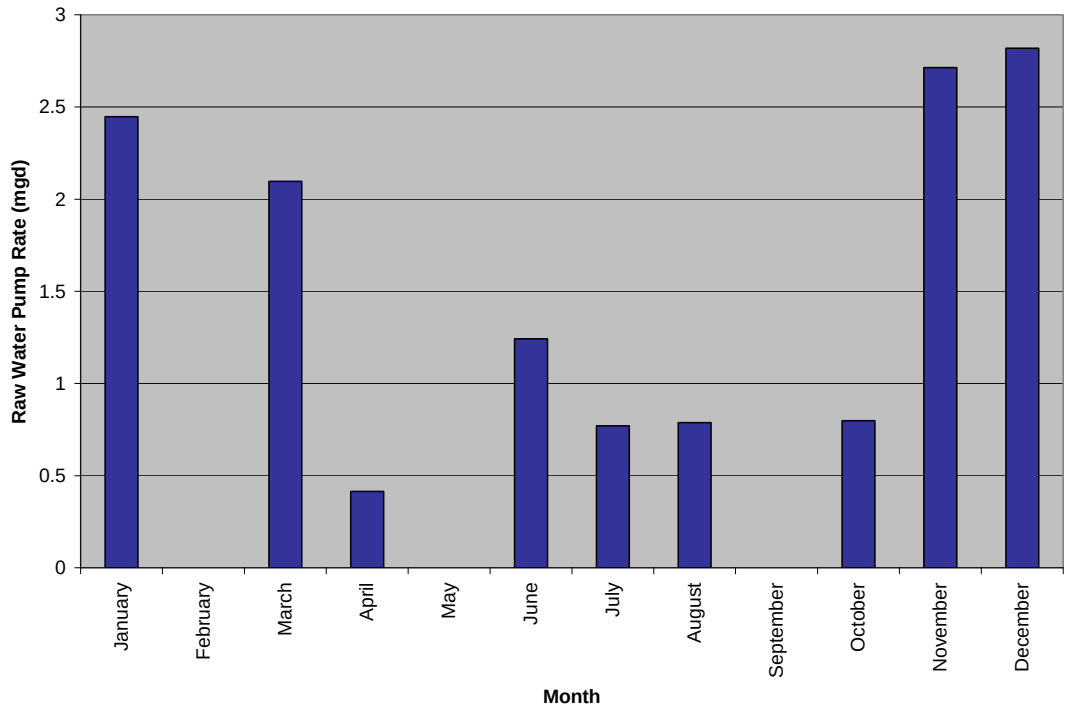


Figure 5. Average Water Pump Rate from the South Cove by Month, 2004-2011

The surface water flow can be further subdivided into dry weather flows (8.41 cfs or 38%) and wet weather flows (13.46 cfs or 62%). Actual flow observed through the pond varies appreciably among seasons and with weather conditions. Additionally, water withdrawals by the Town would also be expected to influence flow rates at Whitman's Pond.

Water withdrawal volumes from Whitman's Pond are summarized in Figure 5. As can be seen, pump rates are highest from November to January. However, water withdrawal is more likely to occur in the months of June and July (Figure 6). It is clear that implementation of management options must be compatible with and scheduled to accommodate water transfer between Whitman's Pond and Weymouth Great Pond for water supply purposes.

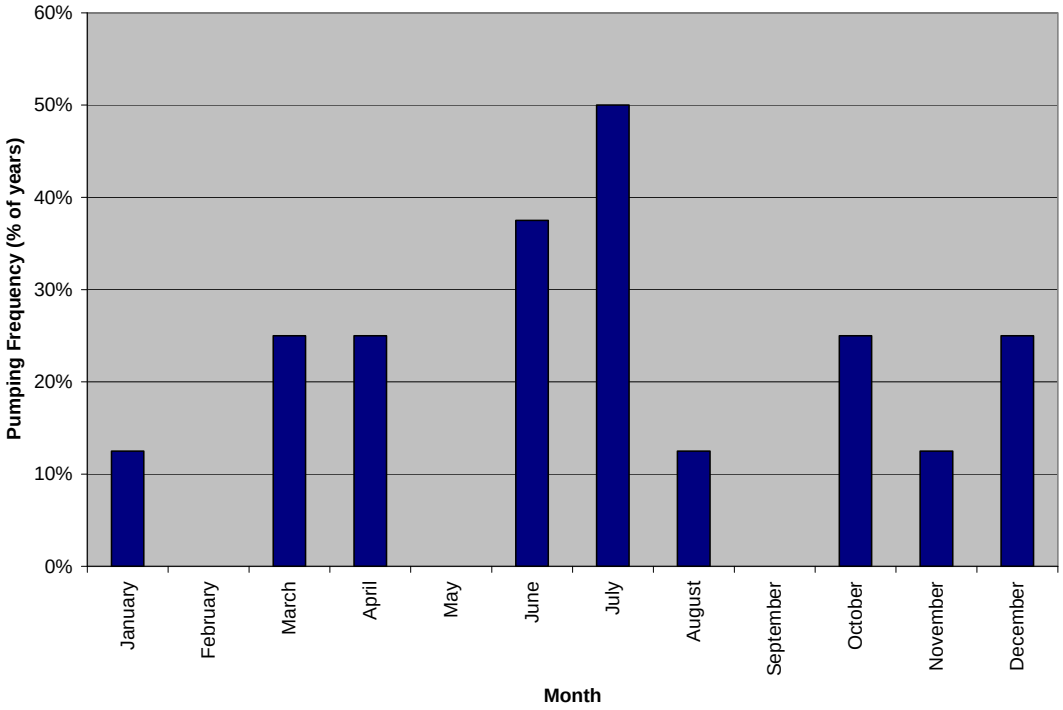


Figure 6. Water Withdrawal Frequency by Month, 2004-2011

Given this hydrologic budget, the estimated detention time for Whitman's Pond averages 41 days, which allows the pond to flush about 9 times per year. In light of the watershed to pond area ratio (approximately 44:1), this flushing rate is not unusual. Implications for pond management would need to be further evaluated for implementation of certain management actions, such as drawdown (see Section 5.2.1).

3.3 Sediment Isopach Mapping and Sediment Quality

The thickest sediments in Whitman's Pond were found at the southern end of the South Cove and in multiple small pockets in the West Cove, where as much as 18.5 feet of soft sediment was found to have accumulated (Table B). Average sediment thickness was close to six feet in both of these coves. Soft sediments in the northwestern area of the Main Basin were thinner, approaching a maximum of 11 feet thick and just over 4 feet on average. The total volume of soft sediments in the mapped portions of Whitman's Pond was estimated to be 625,000 cubic yards, with largest deposits in the South Cove.

Table B. Summary of Key Water and Sediment Dimensions by Basin

Basin	Maximum Water Depth (feet)	Average Water Depth (feet)	Water Volume (millions of gallons)	Maximum Sediment Thickness (feet)	Average Sediment Thickness (feet)	Approximate Sediment Volume (cubic yards)
West	5.0	2.6	14.0	18.5	5.9	150,000
South	7.0	3.3	31.0	16.5	5.8	275,000
Main	26.5	10.2	489.5	10.8	4.2	*200,000

*Only includes the mapped area in the western portion of the Main Basin

Bulk Chemical Results

Bulk chemical sediment data were compared to the Massachusetts Contingency Plan (MCP) S1/GW1 and Beneficial Use Determination (BUD) criteria (Appendix D). Although the MCP standards apply to soil and groundwater, they do provide a basic framework for assessing contaminant concentrations in sediment for a large number of analytes. The MCP S1/GW1 standards were selected because these represent the most stringent category of criteria. BUD thresholds are specific to sediments that may be re-used in the upland environment (e.g., as clean fill or soil amendment). Sediments removed during dredging would be directly compared to the BUD values to determine whether they are acceptable for beneficial uses.

Sediments collected from the Mill River (composite sample MR) showed the greatest number of MCP S1/GW1 exceedances. Heavy metals, including cadmium, chromium, lead, and nickel exceeded MCP S1/GW1 criteria. Arsenic, zinc, and acetone exceeded BUD criteria but were within MCP S1/GW1 criteria. This sample was the only one to produce detectable levels of PCBs (Aroclor 1254 at 480 µg/kg), although total PCBs remained below MCP S1/GW1 criteria.

In the South Cove, MCP S1/GW1 exceedances were limited to SC3, taken from the southern third of the pond. Chromium and nickel were the two analytes exceeding MCP S1/GW1 criteria. Cadmium, lead, zinc, and acetone exceeded BUD criteria. Elsewhere in the South Cove, a few BUD exceedances were measured, including zinc at SC1 and chromium and lead at SC2.

In the West Cove, MCP S1/GW1 criteria were exceeded only for acetone. Cadmium exceeded BUD criteria.

Of the heavy metals found to exceed the MCP S1/GW1 criteria, chromium may not be of significant concern. This is because chromium occurs in two valence states, a trivalent form and a more problematic hexavalent form. Trivalent chromium is an essential element and is considered much less toxic than hexavalent chromium, both for acute and chronic exposure.

Additional sampling for hexavalent chromium would be required to determine whether the observed exceedance was due to this valence state or the less toxic trivalent state.

Bulk Physical Results

Physical testing indicated that pond sediment in the Main Basin was primarily coarse (gravel and sand) with only a small fraction of fines (Table C). Similarly, West Cove sediments were also coarse, with sand being the primary grain size. Sediments in the Mill River and South Cove were dominated by fines, most so in the South Cove at SC1 and SC3. Sand and gravel were present in significant fractions at SC2.

Table C. Whitman's Pond Sediment Sampling Physical Results

Basin	Sample ID	Gravel (%)	Sand (%)	Silt/Clay (%)
Main Basin	MB	61.7	36.1	2.2
Mill River	MR	0.1	35.4	64.5
South Cove	SC1	0.0	15.5	84.5
	SC2	20.2	27.0	52.8
	SC3	0.3	26.6	73.1
West Cove	WC	2.6	92.0	5.4

In general, coarse sediments are less likely to hold onto potential contaminants of concern than fines. Therefore, chemical exceedances in areas where fines make up less than 10% by weight (Main Basin and West Cove) are less problematic than those were fines contribute a significant fraction.

3.4 In-pond Water Quality

In-pond sampling results are presented in Table D. The most striking results are those for dissolved oxygen. Dissolved oxygen in water can be examined in two primary ways: as a concentration or as a percentage of saturation. The amount of oxygen gas that water can hold in solution is a factor of the water temperature; warmer water has a lower saturation point (capable of retaining less oxygen) than colder water. Dissolved oxygen concentrations indicate that the West Cove was experiencing hypoxic conditions during sampling (i.e., dissolved oxygen levels were below the state standard of 5.0 mg/L for support of aquatic life). Dissolved oxygen levels in the South Cove and at the surface of the Main Basin were above state standards for warmwater ponds.

Table D. Water Quality Results from Whitman's Pond, August 29, 2012

Basin	Site ID	Temperature (°C)	DO (mg/L)	DO (%)	Turbidity (NTU)	Color (PCU)	pH (SU)	Total Phosphorus (mg/L)	Total Nitrogen (mg/L)
West Cove	WB	23.7	3.84	45.5	2.56	70	6.2	0.044	*0.605*
Main Basin	MB-S	25.9	6.12	76.3	0.2	60	6.6	0.022	*0.275*
	MB-B	15.1	0.02	0.2	1.97	60	6.4	0.042	1
South Cove	SB	21.0	5.65	72.1	3.37	65	6.5	0.061	1.5

Italics indicate estimate due to one or more analytes not detected at the laboratory quantitation limit.
Secchi Depth = 2.5 m

At the Main Basin station, a profile of dissolved oxygen and temperature was collected between the surface and bottom stations (MB-B and MB-S). The profile demonstrates that the Main Basin was stratified at the time of sampling featuring a warmer, better oxygenated layer (epilimnion) overlaying a cooler, oxygen depleted layer (hypolimnion; Figure 7). A transitional zone known as the metalimnion exists between these two layers. This type of stratified profile is typical for deeper ponds in the summer months. However, in Whitman's Pond the fact that bottom waters are oxygen-starved (anoxic) during the summer means that a large volume of the Main Basin is not supportive of aquatic life.

Total phosphorus levels were elevated (exceeding 0.02 mg/L) at all locations but were most excessive in the South Cove (Table D). Samples collected from the Main Basin showed the lowest phosphorus concentrations, with the surface water sample (MB-S) being much lower than the sample collected from the hypolimnion (MB-B). It is not unusual for bottom waters to have higher levels of phosphorus than surface waters during the summer stratification period. As phytoplankton (algae) grow in surface waters and die off, they remove phosphorus from the epilimnion and carry it with them into the bottom waters, where it may be sequestered, at least temporarily. During pond turnover periods in the autumn and spring, the phosphorus rich bottom waters mix with the depleted surface waters, recharging the water with phosphorus (and other nutrients) that can fuel plankton growth. This is particularly true where water in the hypolimnion is anoxic, a condition that encourages transformation of phosphorus into more soluble forms that can be directly used by plants and algae.

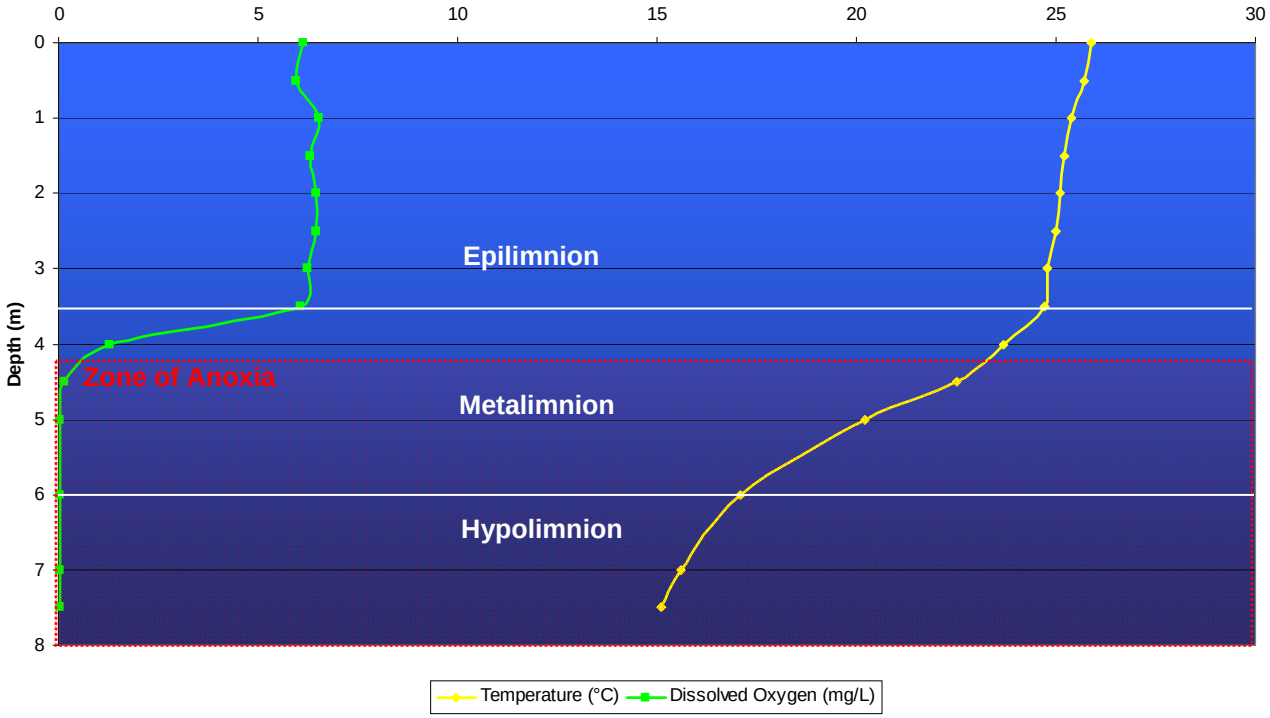


Figure 7. Temperature and Dissolved Oxygen Profiles in the Main Basin of Whitman's Pond, August 29, 2012

Phosphorus is naturally available in relatively small quantities throughout most of New England and is typically considered to be the limiting nutrient in most of our freshwater ecosystems. Some phosphorus is naturally transported to lakes and ponds through atmospheric deposition, wildlife, and runoff from the landscape but this amount is typically small compared to phosphorus generated by human processes. Phosphorus is readily adsorbed onto soil particles, meaning that septic seepage is rarely able to contribute significantly to phosphorus loading into lakes and ponds. However, it is easily transported with soils eroded and transported by runoff from the watershed. Additionally, other sources such as illicit wastewater connections to storm drains, sanitary sewer overflows, or high concentrations of resident waterfowl (e.g., Canada Goose) can also contribute to phosphorus loading. Once phosphorus is in deposited in pond sediments, it may be recycled within the pond through chemical, physical, and biological processes.

Nitrogen is a plentiful nutrient in most fresh water bodies of New England. However, total nitrogen concentrations above 1.0 mg/L may be considered excessive. At Whitman's Pond, elevated levels of nitrogen were evident in the South Cove (Table D). Unlike phosphorus, nitrogen does not readily bind to soil particles. Although natural or background sources of nitrogen exist, such as atmospheric deposition and nitrogen fixation by cyanobacteria, spikes in nitrogen tend to be from human sources, including stormwater runoff and failed septic systems or sewers.

Color, turbidity, and pH measurements at Whitman's Pond were not unusual. The water in the pond is stained, which indicates the presence of dissolved organic carbon. This staining is natural and typical of ponds with extensive wetlands around the shoreline or in the watershed. The West Cove, which is dominated by emergent wetlands at its western end, showed the highest degree of staining (Table D). Turbidity, which measures both dissolved organic carbon and particulate matter (algae, detritus, sediments, etc.) was highest in the South Cove but was not particularly elevated. Lastly, pH, which ranged from 6.2 to 6.6, was well within the typical range for coastal plain ponds in New England.

3.5 Biological Resource Assessment

Fish

ESS reviewed data from fish surveys conducted by the Commonwealth and retained on file by the Westborough field office of the Massachusetts Division of Fisheries and Wildlife (MassWildlife). These fish survey data include records from as early as 1905 with additional surveys from 1956-1958 and 1983, which are summarized in Table E.

The 1905 survey noted that small patches of water lilies were the only substantial aquatic vegetation observed at the time of the survey (mid-September). Sketched maps in the report show that the West Cove, South Cove, and westernmost portion of the Main Basin were not yet flooded at this time. Qualitative descriptions of fish abundance suggest that sunfish species may not yet have been present in the pond.

The surveys from the 1950s documented the presence of alewife, chain pickerel, golden shiner, yellow perch, white perch, brown bullhead, pumpkinseed, and black crappie. Of these, planktivores such as golden shiner and alewife were the dominant fish species.

Banded killifish from Main Basin of Whitman's Pond

Surveys conducted in 1983 found white perch, yellow perch, and alewife at high abundances. Alewife were so abundant that the author even suggested introducing northern pike (*Esox lucius*) or tiger muskie (*Esox masquinongy x Esox lucius*) to Whitman's Pond to control the population.

Rainbow trout (*Oncorhynchus mykiss*) were found in two of the historical surveys (as a stocked exotic game species). However, the history of repeated stocking and the lack of appropriate summer trout habitat (<1% of Whitman's Pond by volume, according the 1983 survey) suggests that rainbow trout are not a self-sustaining species in Whitman's Pond.

Although ESS did not directly observe all of the fish species encountered during previous surveys, the fish species list likely remains similar to what was previously documented (Table F). This is supported by the fish habitat assessment mapping results (Figure 8).

Table E. Summary of Historical Fish Survey Data

Species	Survey Date and Type				
	9/13/1905† Unknown	7/17/1956 Rotenone	7/15/1957 Fyke Net and Rotenone	7/25/1958 Gill Net and Rotenone	7/18-7/19/1983 Electroshock and Gill Net
Alewife	Common	Abundant	Abundant	Abundant	Common
American Eel	Common	Rare	Not Present	Not Present	Not Present
Banded Killifish	Not Present	Not Present	Rare*	Rare	Not Present
Bluegill	Not Present	Uncommon	Abundant	Not Present	Uncommon
Black Crappie	Not Present	Not Present	Rare	Rare	Uncommon
Brown Bullhead	Common	Rare	Common	Uncommon	Common
Chain Pickerel	Abundant	Rare	Common	Common	Rare
Creek Chubsucker**	Not Present	Not Present	Rare	Not Present	Not Present
Golden Shiner	Not Present	Abundant	Abundant	Abundant	Uncommon
Largemouth Bass	Common***	Not Present	Not Present	Not Present	Common
Pumpkinseed	Not Present	Common	Common	Common	Common
Rainbow Trout	Rare	Not Present	Not Present	Not Present	Rare
White Perch	Common	Not Present	Common	Common	Abundant
Yellow Perch	Common	Uncommon	Common	Common	Common

†Abundance assigned based on qualitative data.
*Identified as mummichog, which is primarily a brackish water species unlikely to enter Whitman's Pond.
**Identified as lake chubsucker and noted as an unusual species. Lake chubsucker is not known from New England. However, the closely related creek chubsucker does range into Massachusetts and is sometimes found in lakes (despite the name to the contrary). Therefore, this record was presumed to be creek chubsucker.
***Report refers only to bass – it is assumed that largemouth bass is the species referred to here but this is uncertain, particularly given the apparent absence of largemouth bass in surveys conducted in the 1950s.

These maps indicate that the Main Basin of Whitman's Pond provides habitat for spawning and growth of a wide diversity of fish species (Figure 8). In general, the West Cove and South Cove provide less habitat for species requiring open water but likely support growth and spawning of fish that prefer or tolerate dense weed beds and soft substrates, including predatory brown bullhead and chain pickerel as well as forage fish (banded killifish and golden shiner).

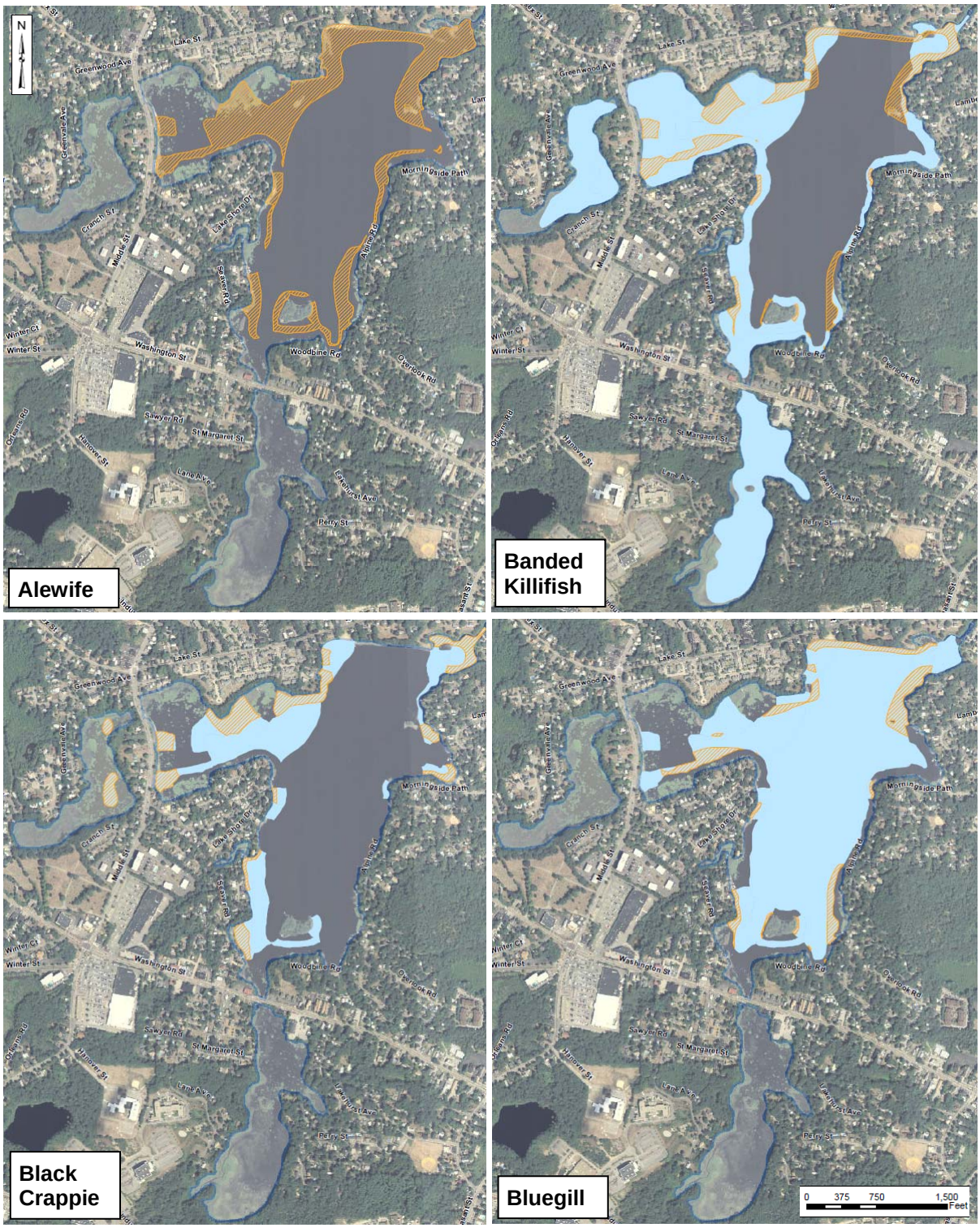


Figure 8a. Most Suitable Fish Spawning and Growth Habitats in Whitman's Pond

Legend

Potentially Suitable Spawning Habitat

Potentially Suitable Growth Habitat

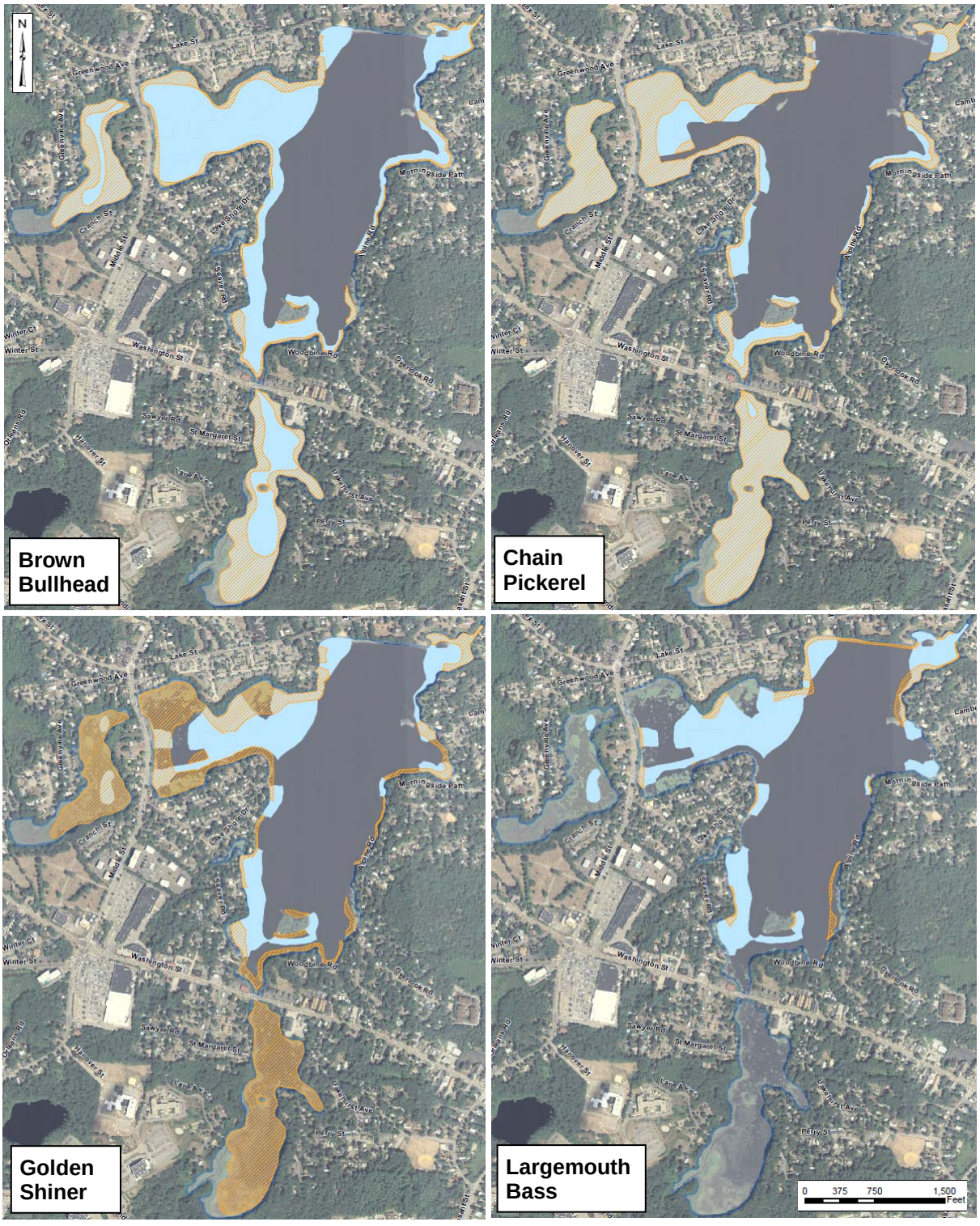


Figure 8b. Most Suitable Fish Spawning and Growth Habitats in Whitman's Pond

Legend

- Potentially Suitable Spawning Habitat
- Potentially Suitable Growth Habitat

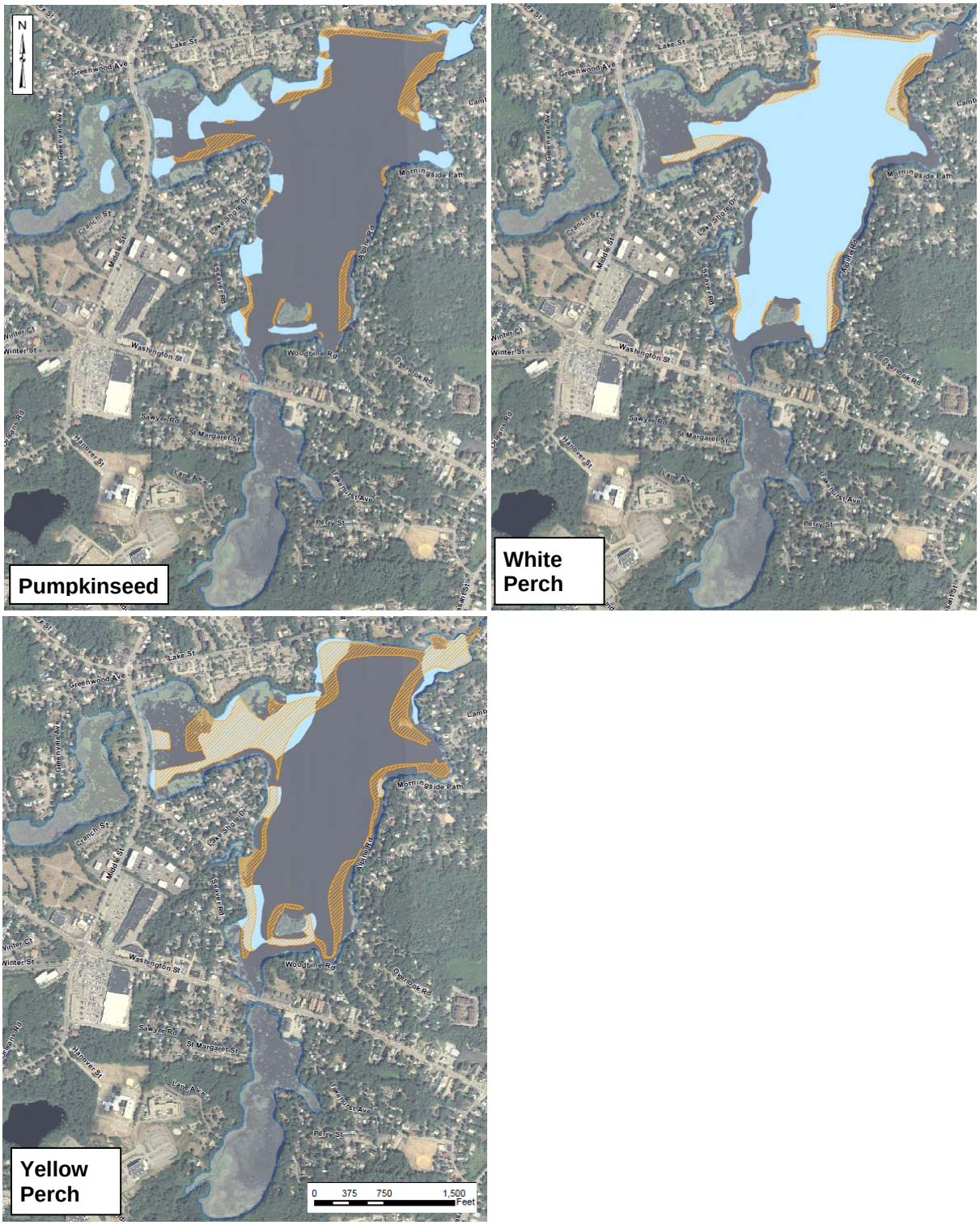


Figure 8c. Most Suitable Fish Spawning and Growth Habitats in Whitman's Pond

The alewife spawning habitat map is presented as a reasonable representation of potential habitats in Whitman's Pond based on observations of alewife habitat use throughout the species range. However, there is a degree of variability in timing and habitat preferences for spawning between different alewife populations and it is possible that local populations are more specific in their habitat preferences. Ideal alewife spawning habitat in Whitman's Pond was hypothesized to be primarily in weed-free sandy areas, such as that currently found on the northeastern shoreline of the Main Basin. However, the fact that this species continues to return in large numbers (approximately 500,000 fish in 2012) despite the continued encroachment of aquatic invasive vegetation suggests that successful alewife spawning may be possible in other habitats (Brad Chase, DMF, personal comm.). At this point, there is not enough evidence to support any particular conclusion and it is likely that the current concept of what constitutes preferred spawning habitat in this population will be further refined in the future.

Habitat for American eel was not specifically mapped as part of this study, due to uncertainty about how long eels remain in the pond as they mature from elvers to yellow eels and finally to the sexually mature silver eel phase (at which point they migrate to the Sargasso Sea for spawning). American eel freshwater habitat preferences range from fine muck (suitable for burrowing) to dense growths of aquatic plants, rocky areas, and large woody debris, which are cumulatively available over large portions of Whitman's Pond. The two factors most likely to restrict eel distribution in the pond are low dissolved oxygen (hypoxia) and, potentially, the presence of flow barriers between the Main Basin and West Cove.

Together these results indicate that Whitman's Pond is capable of supporting a diverse warmwater fish community, as well as spawning habitat for anadromous alewife.

Table F. Fish Species Currently Likely to Inhabit Whitman's Pond*

Common Name	Scientific Name	Date Observed	Basin	Status
Alewife	*Alosa pseudoharengus*	**	NA	Anadromous
American Eel	*Anguilla rostrata*	**	NA	Facultative Catadromous
Banded Killifish	*Fundulus diaphanus*	2	Main	Fresh – warmwater
Black Crappie	*Pomoxis nigromaculatus*	**	NA	Fresh – warmwater
Bluegill	*Lepomis macrochirus*	1, 2	Main	Fresh – warmwater
Brown Bullhead	*Ameiurus nebulosus*	1	NA	Fresh – warmwater
Chain Pickerel	*Esox niger*	1	West	Fresh – warmwater
Golden Shiner	*Notemigonus crysoleucas*	**	NA	Fresh – warmwater
Largemouth Bass	*Micropterus dolomieu*	**	NA	Fresh – warmwater
Pumpkinseed	*Lepomis gibbosus*	1	Main	Fresh – warmwater
White Perch	*Morone americana*	**	NA	Fresh – warmwater
Yellow Perch	*Perca flavescens*	**	NA	Fresh – warmwater

*Excludes vagrants as well as species maintained primarily through stocking (e.g., rainbow trout).
**Not directly observed by ESS but known to inhabit Whitman's Pond based on MassWildlife data.
1. June 7, 2012
2. September 24, 2012

Aquatic Plant Assessment

Nineteen aquatic plant species were observed in Whitman's Pond (Table G), including three exotic invasive species: curly-leaf pondweed (*Potamogeton crispus*), fanwort (*Cabomba caroliniana*) and variable-leaf milfoil (*Myriophyllum heterophyllum*). Each of the exotic species was reported by previous studies – no new species were found in Whitman's Pond. Filamentous green algae was also common in all three basins of the pond. The highest number of aquatic species (12) was observed in the Main Basin while the lowest (7) was observed in the South Cove.

Although not the primary target of the plant mapping efforts, several emergent plant species were also observed around the periphery of Whitman's Pond (Table G). While most of the species observed are generally considered to be beneficial, notable exotic invasive species included common reed (*Phragmites australis*) and purple loosestrife (*Lythrum salicaria*), both of which were well-established at Whitman's Pond. Yellow flag iris (*Iris pseudacorus*), another exotic invasive species, was also observed in widely scattered clumps along the shoreline in the South Cove.

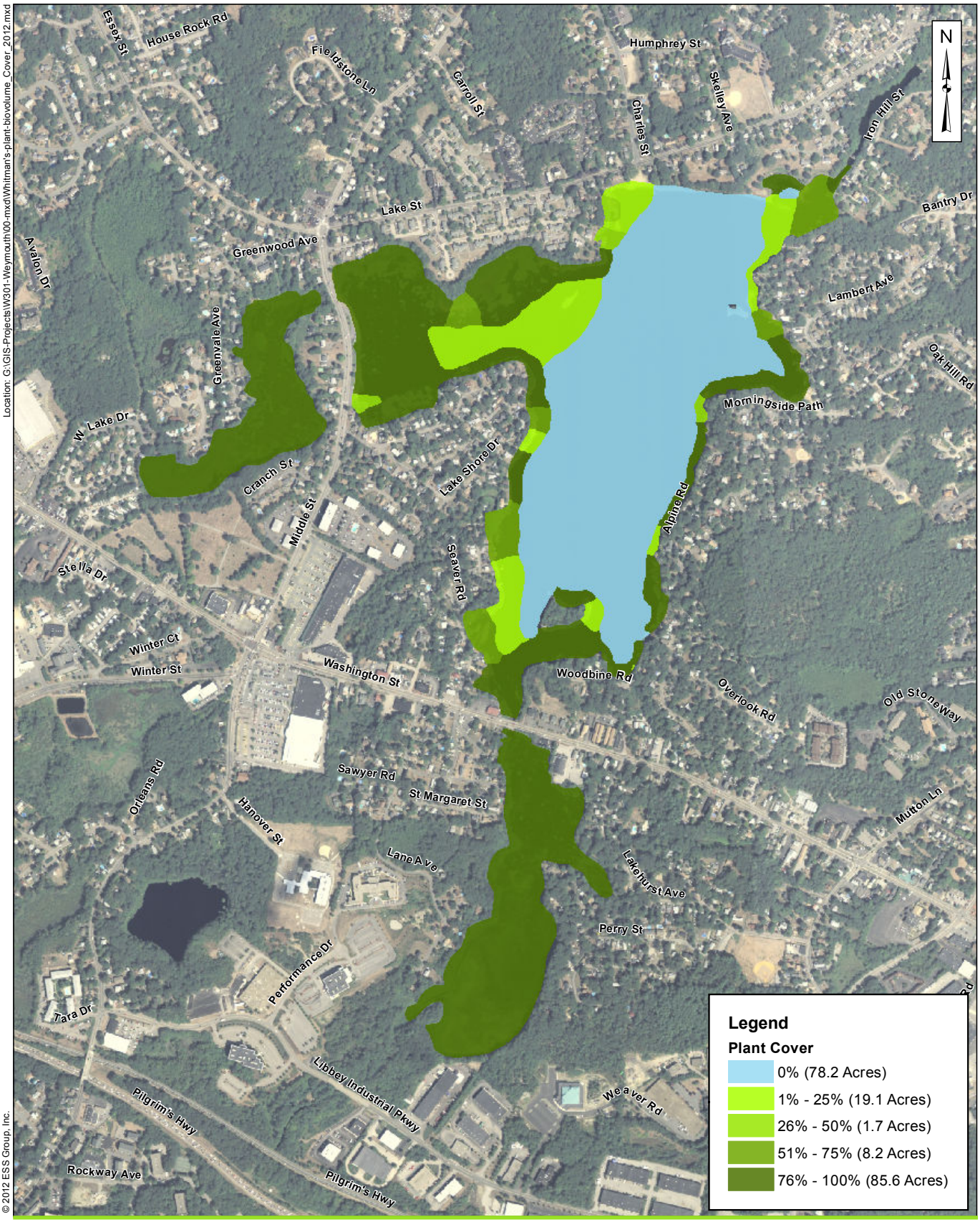
Table G. Aquatic and Emergent Plant Species Observed in Whitman's Pond

Common Name	Scientific Name	Basin	Status
Aquatic Species			
Big Pondweed	*Potamogeton amplifolius*	S	Native
Bladderwort	*Utricularia sp.*	W	Native
Common Bladderwort	*Utricularia macrorhiza*	W	Native
Coontail	*Ceratophyllum demersum*	M, S, W	Native
Curly-leaf Pondweed	*Potamogeton crispus*	W	Exotic Invasive
Duckweed	*Lemna minor*	W	Native
Fanwort	*Cabomba caroliniana*	M, S, W	Exotic Invasive
Filamentous Green Algae	Chlorophyceae	M, S, W	Native
Floating-leaf Pondweed	*Potamogeton epihydrus*	M	Native
Humped Bladderwort	*Utricularia gibba*	M	Native
Little Floating Bladderwort	*Utricularia radiata*	M	Native
Little Floating Heart	*Nymphoides cordata*	W	Native
Thinleaf Pondweed	*Potamogeton pusillus*	M	Native
Variable-leaf Milfoil	*Myriophyllum heterophyllum*	M, S	Exotic Invasive
Water Celery	*Vallisneria americana*	M	Native
Water Starwort	*Callitriche heterophylla*	S	Native
Watershield	*Brasenia schreberi*	M	Native
Western Waterweed	*Elodea nuttalii*	M	Native
White Water Lily	*Nymphaea odorata*	M, S, W	Native
Yellow Water Lily	*Nuphar lutea variegata*	M, S, W	Native

Table G. Aquatic and Emergent Plant Species Observed in Whitman's Pond

Common Name	Scientific Name	Basin	Status
Emergents			
Burreed	*Sparganium sp.*	M	Native
Cattail	*Typha latifolia*	M, W	Native
Common Reed	*Phragmites australis*	M, W	Exotic Invasive
Pickerelweed	*Pontederia cordata*	M, S, W	Native
Purple Loosestrife	*Lythrum salicaria*	M, S, W	Exotic Invasive
Sedge	*Carex spp.*	M	Native
Water Smartweed	*Polyganum sp.*	M, W	Native
Water Willow	*Decodon verticillatus*	M, S	Native
Woolgrass	*Scirpus cyperinus*	W	Native
Yellow Flag Iris	*Iris pseudacorus*	S	Exotic Invasive

Aquatic plant cover was very dense (greater than 75%) throughout the West Cove and South Cove (Figure 9). The western and southern portions of the Main Basin were also characterized by extensive areas of very dense plant cover. All together, nearly 86 acres of Whitman's Pond were characterized by very dense plant cover. However, large areas of little to no aquatic plant cover extended over much of the central portion of the Main Basin, even into some shallow water areas on the northern end.



VEGETATION MANAGEMENT ACTION PLAN
Whitman's Pond, Weymouth, Massachusetts

Aquatic Plant Cover

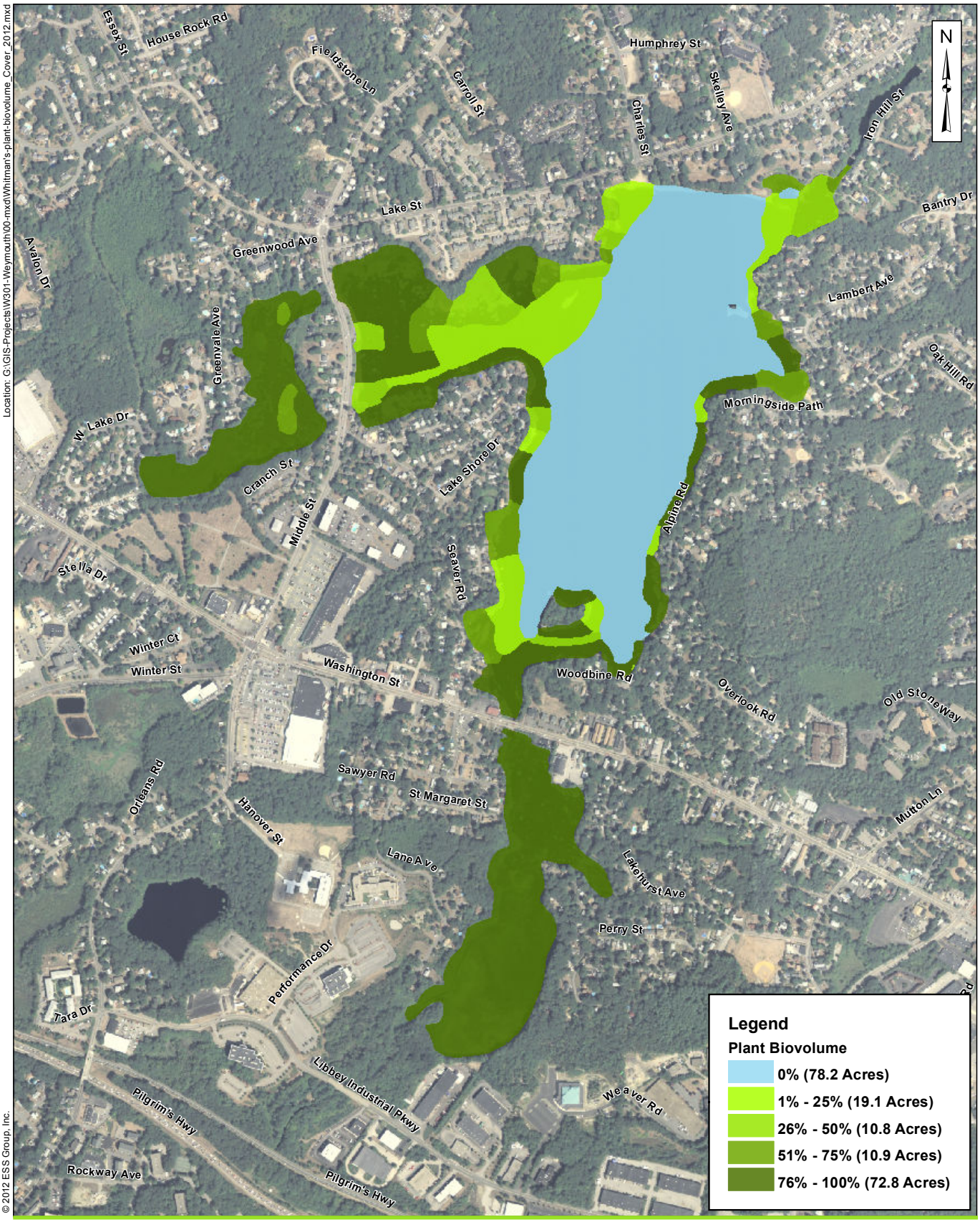
ess group
environmental consulting & engineering services

Scale: 1" = 1,000'

0 1,000 Feet

Source: 1) ESS Plant Biovolume Data, 2012
2) NAIP Orthophotography, 2009

Figure 9



Location: G:\GIS-Projects\W301-Weymouth\00-mxd\Whitman's-plant-biovolume_Cover_2012.mxd

VEGETATION MANAGEMENT ACTION PLAN
Whitman's Pond, Weymouth, Massachusetts

Aquatic Plant Biovolume

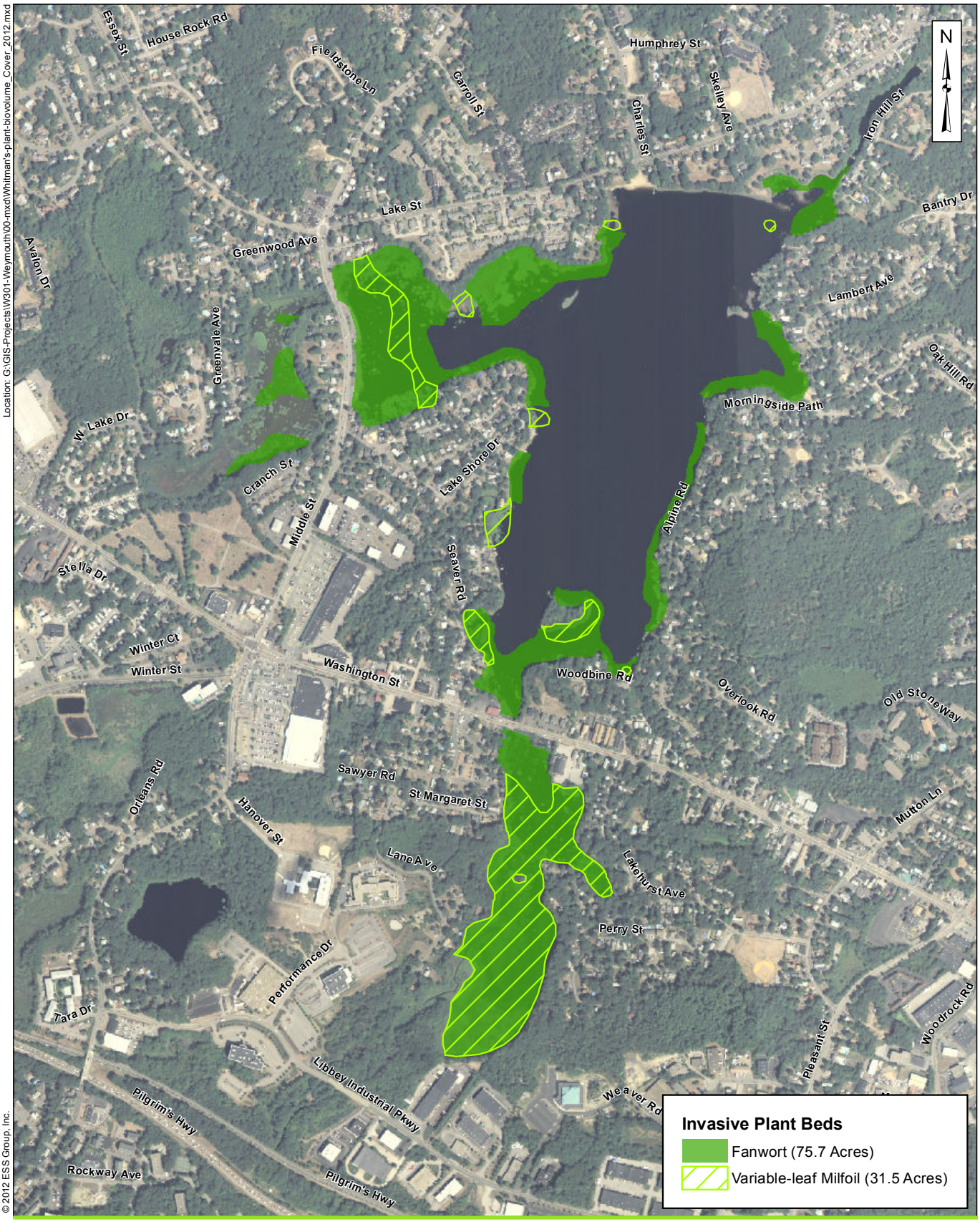
ess group
environmental consulting & engineering services

Scale: 1" = 1,000'

0 1,000 Feet

Source: 1) ESS Plant Biovolume Data, 2012
2) NAIP Orthophotography, 2009

Figure 10



VEGETATION MANAGEMENT ACTION PLAN
Whitman's Pond, Weymouth, Massachusetts

Aquatic Invasive Plant Beds

ess group
environmental consulting & engineering services

Scale: 1" = 1,000'

0 1,000 Feet

Source: 1) ESS Plant Biovolume Data, 2012
2) NAIP Orthophotography, 2009

Figure 11

Aquatic plant biovolume, which is defined as the percentage of the water column occupied by plants, generally mimicked patterns in plant cover and biovolume was very high (i.e., greater than 75%) over nearly 73 acres of the pond (Figure 10). However, small areas of lesser biovolume were present in the West Cove and to a greater extent in the Main Basin. These areas of reduced plant biovolume, whether natural or maintained by boating activity, allow more access to recreational boats and may provide limited edge habitat and open water transit corridors for fish during the peak season of plant development. Conversely, the South Cove did not appear to contain any significant areas of lesser biovolume. Rather, growth there consisted of more or less continuous aquatic plant beds extending from the pond bottom to the water surface.

Fanwort grows to the surface in dense beds across much of the pond.

Fanwort and variable-leaf milfoil were the most commonly observed plants pondwide, often forming extensive beds (Figure 11). In the South Cove, fanwort beds essentially extended across the entire basin and were frequently accompanied by variable-leaf milfoil and water lilies. The West Cove also contained large fanwort beds, although water lilies were dominant. Fanwort beds ringed the Main Basin, with the densest and most extensive growths in the western and far southern portions of the basin. Variable-leaf milfoil beds also occurred in scattered patches around the Main Basin.

Curly-leaf pondweed was not observed in sizable beds at the time of survey. In fact, it was only recorded at one location in the West Cove. However, this species tends to reach maximum development in late May to early June and quickly dies back after that time. Therefore, it may have been underrepresented in our aquatic plant survey, which was conducted in the late summer period.

Aquatic Macroinvertebrates

Twenty-two aquatic macroinvertebrate taxa were observed in the four sampling areas of Whitman's Pond (Table H). Native freshwater mussels including the eastern elliptio (*Elliptio complanata*) and eastern floater (*Pyganodon cataracta*) were found in silty and sandy habitats of the Main Basin. Other freshwater taxa, including snails, clams, crustaceans, aquatic mites, aquatic worms and aquatic insects were observed.

Eastern elliptio mussels were common in the Main Basin.

Of note, two exotic species were encountered: Asian clam (*Corbicula fluminea*) and Chinese mystery snail (*Cipangopaludina chinensis*). Asian clam appeared to be most common on the northern end of the Main Basin while Chinese mystery snail was more widespread throughout the pond. However, both infestations appear to be well-established and would be difficult to eradicate. These species can become a minor nuisance by contributing to problems such as blockage of water intake structures. Their impact on pond ecology is not well-documented but is likely to be minor. Given these factors, control actions beyond monitoring and maintenance of water intakes are not currently recommended.

Table H. Aquatic Macroinvertebrates Observed at Whitman's Pond on September 25, 2012

Group	Common Name	Scientific Name	Monitoring Area	Status
Clams and Mussels	Asian Clam	*Corbicula fluminea*	2	Exotic
	Eastern Elliptio	*Elliptio complanata*	2	Native
	Eastern Floater	*Pyganodon cataracta*	2, 3	Native
Aquatic Snails	Chinese Mystery Snail	*Cipangopaludina chinensis*	2, 3, 4	Exotic
	Pouch Snail	*Physa acuta*	1, 4	Native
	Ram's Horn Snail	Planorbidae	1, 4	Native
Crustaceans	Aquatic Sowbug	*Caecidotea communis*	2	Native
	Crayfish	*Orconectes sp.*	4	Native
	Scud	*Hyallela azteca*	1, 2, 3, 4	Native
Aquatic Mites	Aquatic Mite	Hydrachnidia	3	Native
Flatworms	Flatworm	Planaria	3, 4	Native
Aquatic Worms	Leech	*Helobdella stagnalis*	3	Native
	Tubificid Worm	Tubificidae	1	Native
Damselflies	Bluet	*Enallagma spp.*	2, 3	Native
	Forktail	*Ischnura spp.*	2, 4	Native
Dragonflies	Dragonfly	Corduliidae	3, 4	Native
Mayflies	Angler's Curse Mayfly	*Caenis sp.*	4	Native
	Blue-winged Olive Mayfly	Baetidae	4	Native
	March Brown Mayfly	*Maccaffertium sp.*	3	Native
True Bugs	Water Boatman	Corixidae	4	Native
Caddisflies	Tubenet Caddisfly	Polycentropodidae	2	Native
Beetles	Crawling Water Beetle	Haliplidae	4	Native
True Flies	Non-biting Midge	Chironomidae	2, 3, 4	Native
	True Fly	Brachycera	4	Native

Other Species

Whitman's Pond appears to provide sufficient habitat for common reptiles, amphibians, birds and furbearer mammals.

The herpetofauna (reptiles and amphibians) observed by ESS in Whitman's Pond include common, widespread species (Table I). No rare species were observed. However, this list is not intended to be exhaustive and other common herpetofauna are likely to use Whitman's Pond or its immediate surroundings. Woody debris, exposed boulders, and floating plant beds provided plenty of daytime basking habitat for painted turtle and other turtle species active during the day. Appropriate habitat for musk turtle and green frog (soft bottom ponds with plant cover) was also plentiful.

Musk turtle is a common but secretive species observed at Whitman's Pond

Canada Goose was the most frequently observed avian species

Table I. Herpetofauna Observed Using Whitman's Pond

Common Name	Scientific Name	Dates Observed	Basin
Painted Turtle	*Chrysemys picta*	1, 2, 3	S, W
Musk Turtle	*Sternotherus odoratus*	4	M
Green Frog	*Rana clamitans*	1, 2, 3	M, S, W

1. June 7, 2012
2. July 19, 2012
3. August 29, 2012
4. September 24, 2012

ESS observed 22 avian species using the pond or its immediate shoreline during our five field visits (Table J). This list is not exhaustive and many additional avian species would be expected to use the pond and immediate surroundings for one or more periods of the year. During our field visits, Canada Goose (*Branta canadensis*), Belted Kingfisher (*Megaceryle alcyon*), Mute Swan (*Cygnus olor*), and Herring Gull (*Larus smithsonianus*) were the most frequently observed species, although Canada Goose and Herring Gull were the numerically dominant species. Canada Goose was observed in flocks large enough to be a public nuisance to traffic on Middle Street and visitors to the adjacent public shoreline area. Large numbers of goose droppings were observed along this area of shoreline and in nearshore waters during each field visit.

Table J. Avian Species Observed Using Whitman's Pond and Shoreline Habitats

Common Name	Scientific Name	Dates Observed	Basin
American Robin	*Turdus migratorius*	3	M, S
Belted Kingfisher	*Megaceryle alcyon*	2, 3, 4, 5	M, W
Black-crowned Night-heron	*Nycticorax nycticorax*	2	M
Blue Jay	*Cyanocitta cristata*	2, 5	S, M
Canada Goose	*Branta canadensis*	1, 2, 3, 4, 5	M, W
Common Grackle	*Quiscalus quiscula*	2, 3	S

Table J. Avian Species Observed Using Whitman's Pond and Shoreline Habitats

Common Name	Scientific Name	Dates Observed	Basin
Common Yellowthroat	*Geothlypis trichas*	3	M, S
Cooper's Hawk	*Accipiter cooperii*	3	M
Double-crested Cormorant	*Phalacrocorax auritus*	2, 3	S
Eastern Kingbird	*Tyrannus tyrannus*	2, 3	M, W
Eastern Phoebe	*Sayornis phoebe*	3	M, S
Gray Catbird	*Dumetella carolinensis*	3	M, S
Great Black-backed Gull	*Larus marinus*	3	M, S
Great Blue Heron	*Ardea herodias*	3, 4	M, W
Great Egret	*Ardea alba*	1, 2	M, W
Herring Gull	*Larus smithsonianus*	2, 3, 5	M
Mallard	*Anas platyrhynchos*	3, 4	M, S
Mute Swan	*Cygnus olor*	1, 2, 3, 4	M, W
Northern Cardinal	*Cardinalis cardinalis*	3	M, S
Osprey	*Pandion haliaetus*	2, 3	M, W
Red-shouldered Hawk	*Buteo lineatus*	5	M
Red-winged Blackbird	*Agelaius phoeniceus*	2	W, S

1. April 2, 2012
2. June 7, 2012
3. July 19, 2012
4. August 29, 2012
5. September 24, 2012

Athough no furbearers were directly observed during the field program, habitat for muskrat (*Ondatra zibethicus*) appears to be available at Whitman's Pond. Additional limited habitat for beaver (*Castor canadensis*) and river otter (*Lontra canadensis*) may also be available. Of these species, muskrat has been previously documented from the Weymouth Back River ACEC downstream (Meyers 1997).

Wetland Assessment

Whitman's Pond has a mostly narrow and incomplete fringe of bordering vegetated wetland along its shoreline. Emergent wetlands are extensive on the shallow western end of the West Cove and southern end of the South Cove. An area of scrub-shrub/emergent wetlands is present just south of Memorial Drive along a small portion of the northern shore of the Main Basin.

Scrub-shrub (left) and emergent (right) wetlands are present over limited areas

Overall, Whitman's Pond provides valuable fish and wildlife habitat through the various wetland and open water habitats that occur within the pond. A species-rich assemblage of fish, birds, invertebrates, and plants is still supported by Whitman's Pond as a whole. A few species of herpetofauna and mammals also appear to be present. However, the expansion of invasive plants, particularly exotic aquatic species, has resulted in large areas being dominated by beds of one or two species of high biovolume aquatic plants. These beds have encroached upon habitats that might otherwise be open water or inhabited by a more diverse matrix of native species. Additionally, resident populations of Canada Goose appear to present a safety and health risk to the public at Whitman's Pond.

3.6 Recreational Use

Whitman's Pond is directly accessible to many of the residents living along the shoreline of the Main Basin and South Cove. Access to open water from private properties on the West Cove is generally better from the central and eastern shorelines, as much of the original western shoreline converted to emergent wetland.

Public access to the West Cove at Greenvale Avenue

Despite the large number of private properties abutting the Whitman's Pond shoreline, public access is relatively well-developed. Improved public access locations are present at two locations on the West Cove and two on the Main Basin. Additional primitive public access exists at multiple locations (particularly road crossings) on the shoreline of each of these basins. The South Cove is also publicly accessible from the western shoreline at the end of Echo Avenue.

Whitman's Pond is currently used for a wide variety of recreational activities. These include, but are not necessarily limited to fishing, boating (motorized and non-motorized), wildlife observation, ice skating, and passive recreation. A new informational kiosk was installed near the boat ramp on the western end of the Main Basin in December 2012.

4.0 MANAGEMENT GOALS

The management goals for Whitman's Pond have been clearly defined by Whitman's Pond Working Group and include protection or improvement of the following:

- Town water supply (quality and quantity)
- Recreational opportunities
- Ecological value, particularly with regard to alewife spawning habitat

Given the number of issues currently impacting Whitman's Pond, including excessive aquatic weed growth, sedimentation, and general eutrophication, a wide range of management options were contemplated.

A feasibility assessment of potential management options with respect to achieving the management goals above is presented in the following section.

5.0 MANAGEMENT OPTIONS: FEASIBILITY ASSESSMENT AND RECOMMENDATIONS

This section presents the range of options for vegetation management in Whitman's Pond, based on the goals stated in Section 4.0. Summaries of each option are presented in Table K and cross-referenced to the appropriate subsection, where the feasibility assessment and recommended options are treated in

greater detail. Summary tables of suggested timeline and estimated costs for all recommended options are available in Appendix E.

Table K. Summary Table of Management Options for Whitman's Pond

Approaches	Issue(s) Addressed	Primary Pros	Primary Cons	Cross-reference
Short-term Options for Consideration*				
Partial Lake Systemic Herbicide (Fluridone)**	Fanwort control	Works quickly and provides control for two or more seasons	• Long contact time and extent of fine sediments may result in need for reapplications • Best time for application conflicts with alewife reproduction • Does not work well on variable-leaf milfoil • High cost (for herbicide)	Section 5.1.1
Partial Lake Contact Herbicide (Flumioxazin)**	Fanwort and variable-leaf milfoil control	• Works quickly against multiple species with minimal contact time • Can be applied outside of the alewife spawning period	• Must be done in phases to avoid extreme DO crashes • Only provides one season of control • Applications outside of the alewife spawning window may allow plants to grow unchecked over early part of the peak recreational season • High cost (for herbicide)	Section 5.1.1
Hand Harvesting	Control of small shoreline infestations	Can be done by volunteers	Labor intensive with minimal areal impact	Section 5.1.5
Diver Assisted Suction Harvesting (DASH)	Control of small infestations or isolated beds as follow-up to other treatment	More efficient and less likely to spread fragments than hand harvesting	Labor intensive and costly over larger areas	Section 5.1.5
Biological Control (Loosestrife Beetles)	Purple loosestrife control	Inexpensive with no impact to desirable native species	• Population requires time and contiguous areas of purple loosestrife to become established • Control only – eradication not feasible	Section 5.1.2
Bottom Sealing (Benthic Barriers)**	Local macrophyte control	Immediately effective in eliminating macrophyte growth	Numerous drawbacks, most notably the high cost. Best over very small areas near docks and beaches (<1 acre).	Section 5.1.3

Table K. Summary Table of Management Options for Whitman's Pond

Approaches	Issue(s) Addressed	Primary Pros	Primary Cons	Cross-reference
Resident Waterfowl Control	Phosphorus and bacteria loading reduction	• Removes a source of nutrients and bacteria • Prevents wildlife conflicts on Middle Street and at public access	• Active control methods may be controversial • Passive control methods may be expensive or unattractive	Section 5.1.6
Hydroraking or Rotovation**	Water lily control	Best way to quickly control nuisance growths of water lilies and create open water habitat	Encourages spread of vegetatively reproducing species	Section 5.1.4
Long-term Options for Consideration*				
Drawdown**	Shallow-water macrophyte control in Main Basin/South Cove	May achieve good control in shallow waters at minimal operating cost	• Effectiveness limited by weather • Reduces or eliminates winter recreation activities and fish habitat • May impact downstream waters	Section 5.2.1
Dredging**	Shallow water depth Thick sediment deposits Overall macrophyte control	Addresses multiple in-pond problems and lasts for decades	• More lengthy permitting process • Reduces or eliminates access to the pond during dredging • High initial cost – possibly made higher by presence of pollutants	Section 5.2.2
Control Nutrient and Sediment Loading	Water quality	Addresses underlying problems at the source (i.e., in the watershed)	• Does not address internal (in-pond) recycling of nutrients • Whitman's Pond watershed spans municipal boundaries – further improvements in water quality will require time, capital and maintenance expenses, and watershed-level coordination	Section 5.2.3
Other Recommended Approaches	Prevention of future pond management problems	Results in a better informed public Costs are minimal	Few potential drawbacks	Section 5.2.4

Table K. Summary Table of Management Options for Whitman's Pond

Approaches	Issue(s) Addressed	Primary Pros	Primary Cons	Cross-reference
Monitoring and Reporting	Water quality Ecosystem function Invasive species	Documents and assesses watershed and in-pond problems Documents success or failure of management actions May provide opportunity for community involvement	Does not directly provide management action	Section 6.0
Options Assessed but not Currently Appropriate				
Herbicides (Excluding Fluridone and Flumioxazin)**	See text	See text	See text	Section 5.1.1
Biological Controls (Excluding loosestrife beetles)				Section 5.1.3
Mechanical Harvesting**				Section 5.1.5
Plant Competition				Section 5.3.1
Dilution or Flushing				Section 5.3.2
Hypolimnetic Withdrawal				Section 5.3.2
Shading Dye**				Section 5.3.3
Nutrient Inactivation				Section 5.3.4
Aeration and/or Destratification				Section 5.3.5
Novel Approaches (e.g., Limno-barriers)				Section 5.3.6

*A summary of timelines and costs for all recommended options is available in Appendix E.
**Indicates non-selective methods with potential for some impact to non-target organisms.

5.1 Recommended Short-term Options

Short-term management options include those that either require significant planning or have an effective period of five years or less. Some short-term management options (e.g., chemical treatment) may involve short-term actions implemented repeatedly to achieve long-term management goals. However, significant cost or effort is required each time the option is implemented.

5.1.1 Chemical Treatment (Herbicides) – Recommended for Partial-lake and Spot Treatment Only

Herbicides remain a controversial aquatic weed control measure in many communities because of their association with pesticides, which is generally perceived to be negative. However, as we learn more

about the various negative impacts that can be associated with alternative physical and biological management options, chemical control measures continue to be used as part of many balanced lake management plans.

Although no herbicide is completely safe or harmless, a premise of federal pesticide regulation is that the potential benefits derived from use outweigh the risks when registered herbicides are applied according to label recommendations and restrictions. Current herbicide registration procedures are far more rigorous than in the past and the ability of qualified and licensed applicators to target applications of herbicides further improves the relative safety of using these chemicals for nuisance aquatic plant control. However, each of the herbicides evaluated in this Vegetation Management Action Plan present some degree of risk with regard to potential toxicity to non-target organisms and temporary recreation or water withdrawal restrictions would be needed for herbicide applications at Whitman's Pond. Restrictions vary by herbicide formulation. However, restrictions on fishing and other non-contact recreation are generally not required.

In Whitman's Pond, herbicides present an additional challenge due to the timing of the annual river herring spawning run, which coincides with the ideal time to apply systemic herbicides for maximized uptake by target species. Given the undocumented species-specific effects of herbicides on alewife eggs and larvae as well as the potential for dissolved oxygen depressions from treated beds, pondwide herbicide treatments were denied by the Weymouth Conservation Commission in May 2010. In light of the significant concerns with river herring, ESS has discounted pondwide treatment as a viable option at this time and we recommend that any herbicide applications be limited to partial pond or spot treatments.

In the short-term, herbicide treatment is one of the most cost-effective means by which to rapidly achieve the goal of reducing aquatic weed biomass over a large area. Herbicides may also be used over the long-term as part of a comprehensive management plan to treat areas of recurring infestations that are not readily controllable through other means (e.g., where a winter drawdown is used to manage most invasive weed growth with herbicides selectively applied to areas deeper than the drawdown can impact).

Herbicides can only be applied by state-licensed herbicide applicators. Therefore, this is not an option that pond residents or volunteers can undertake themselves. Costs for permitting an herbicide treatment are typically low ($3,500 to $5,000) because a Notice of Intent to the local Conservation Commission is all that is usually required. Fluridone (Sonar) treatment is already permitted under the existing Order of Conditions but treatment with other types of herbicides could require an amended Order of Conditions. Addditionally, associated monitoring expenses will likely also be required to ensure any treatments within the time-of-year restriction proposed by Massachusetts DMF in their May 26, 2010 letter are protective of alewife spawning at Whitman's Pond. The cost of monitoring associated with an herbicide treatment will vary greatly depending on any special conditions imposed in the Order of Conditions. A basic monitoring program involving pre-treatment, during treatment, and post-treatment monitoring of dissolved oxygen levels, non-target organisms (e.g., fish), and aquatic habitat could be implemented for a cost of approximately $4,500 to $6,000, depending on extent and intensity of sampling required.

The two herbicides currently recommended for use at Whitman's Pond are fluridone (trade name Sonar) and, pending approval for use in Massachusetts, flumioxazin (trade name Clipper). The method of action, contact time, and target species vary by herbicide and it is likely that more than one formulation would need to be used at some point to achieve desired results. More detail on the usage and costs of each of these is provided in the following sections.

Fluridone – Systemic Herbicide: Fluridone is a systemic herbicide that works by inhibiting the production of phytoene desaturase, an enzyme involved in the production of carotenes. Interrupting this process eventually leads to reduced photosynthesis and starvation of the plant. However, fluridone concentrations must be maintained at treatment levels for three weeks to a month (sometimes longer) to

achieve effective treatment. Due to the slow action of this herbicide, plant dieback is gradual and dissolved oxygen sags are rarely problematic.

Fluridone is highly effective on fanwort but less so on variable-leaf milfoil. Therefore, control of variable-leaf milfoil, which grows at lower densities and extents in Whitman's Pond, may require additional management with another herbicide or management technique. Post-treatment monitoring should allow the effect of treatment on variable-leaf milfoil to be quantified so that additional management action can be taken, if necessary.

One drawback of fluridone is that it is best applied relatively early in the growing season (usually June) to maximize uptake. This conflicts with the critical April to June time period for river herring reproduction in Whitman's Pond. It is possible to treat outside of this window but the control achieved may be reduced.

Fluridone may be applied as a liquid formulation (requires multiple treatments to maintain high concentration) or as a slow-release pellet formulation. The pellet formulation can produce good results but is subject to burial (which can result in poor control) in soft silts and mucks. In areas where surface sediments are particularly soft (e.g., the South Cove) the liquid formulation may be required to ensure treatment at a sufficient concentration. At additional cost, limno-barriers may be used to maintain treatment concentrations within select treatment areas while reducing impacts to non-target areas (see Section 5.3.6). Lower application rates are recommended within 1,320 ft of a public water intake.

Fluridone is one of the more expensive herbicides available and costs up to $1,000/acre to apply. However, it is usually guaranteed by the applicator for two to three seasons of control. If used for long-term control, this approach would likely need to be repeated every other year or at least every third year.

If desired, a treatment program that involves treating one basin each year could be envisioned to distribute annual costs more evenly.

***Flumioxazin – Contact Herbicide*:** Flumioxazin (trade name Clipper) is a fast-acting contact herbicide that requires only 4 to 6 hours of contact time and can achieve results in less than 24 hours. A primary benefit of flumioxazin is that it is effective against fanwort, variable-leaf milfoil, and curly-leaf pondweed, each of which is known to be present in Whitman's Pond. It can also be used effectively on certain nuisance species of floating-leaf plants.

Flumioxazin has been approved by the US EPA for use in agriculture since 2001 and on aquatic plants since 2010. Since 2010, it has been registered in 47 states including all six New England states. In Massachusetts, flumioxazin (as Clipper formulation) was registered for use in lakes and ponds in 2012. Guidelines for use of were published by the Massachusetts Division of Agricultural Resources (MDAR) and MassDEP in 2013.

Flumioxazin breaks down quickly and does not accumulate in sediments so risks to non-target areas are minimal. Additionally, flumioxazin has very low toxicity to most animal life, including humans and birds, and does not require any post-application restrictions for drinking water or recreation (although the herbicide applicator will likely recommend a brief post-treatment restriction on these activities as standard practice). As with most herbicides, Clipper does have the potential to be toxic to invertebrates and fish. Although toxicity has only been established at concentrations well above the typical treatment concentrations, MDAR/MassDEP guidelines recommend certain restrictions and monitoring until the product's impacts on non-target organism are better understood.

This herbicide works by inhibiting protoporphyrinogen oxidase (PPO), an enzyme necessary for photosynthesis. Inhibition of PPO causes destruction of plant cell plasma membranes in the presence of sunlight, resulting in rapid dieback of plant tissues. As might be expected, plant cells not directly exposed to the agent or sunlight (e.g., roots) are not killed by flumioxazin. Therefore, plants with sufficient energy reserves may re-grow during the subsequent growing season. Flumioxazin can be applied at any time of

the year when the plant is actively growing but works best if applied before too much canopy shading has developed. This may allow for effective applications outside the April to June spawning window for alewife. However, later applications also imply that significant plant growth could occur prior to treatment, thereby reducing the useful period of open water conditions for recreation.

Limno-barriers may be used to control the area of flumioxazin exposure (see Section 5.3.6). This could be a helpful technique for any initial flumioxazin treatments at Whitman's Pond (e.g., for a flumioxazin pilot study) until the impacts of this herbicide on sensitive ecological resources are better understood.

The primary limitation to flumioxazin is that it carries a higher price than most other contact herbicides and can be expected to cost approximately $1,000/acre to apply. Additionally, due to the fast action of flumioxazin, treatment should avoid covering large areas (e.g., an entire basin) in one day to prevent massive amounts of decay and nutrient release all at one time. Such a scenario could potentially result in localized dissolved oxygen depletion or trigger an algal bloom.

ESS recommends incorporating flumioxazin as a method for partial lake treatment with follow-up treatments after initial application of fluridone, or in tandem with other non-herbicide management actions. Flumioxazin treatments should focus on controlling smaller growths of invasive aquatic plants, particularly spots where variable-leaf milfoil and fanwort are known to grow in mixed beds.

Other Herbicide Options – Not Currently Recommended

Other herbicides are available to treat nuisance vegetation in Whitman's Pond. However, they are not currently recommended, as they are not effective on fanwort, the primary nuisance species in Whitman's Pond. Additionally, each of these herbicides has additional limitations that make them less desirable for use at this time. A brief description of each of these is provided below.

***Diquat dibromide – Contact Herbicide*:** As a contact herbicide, diquat (trade name Reward) works by interrupting the photosynthetic process, resulting in the dieback of leaf and stem cells. It offers immediate control of variable-leaf milfoil growth and is one of the least-expensive approved herbicides available (typically less than $300/acre). However, this control would only be expected to last one season as diquat does not effectively kill rooted portions of aquatic vegetation. Furthermore, while not effective on fanwort, diquat is non-selective and would likely impact a broad spectrum of native plants. Each of these drawbacks reduces the apparent cost-effectiveness of diquat over the long term. Therefore, diquat is not currently recommended.

***Triclopyr – Systemic Herbicide*:** Triclopyr (trade name Renovate) is a dicot-selective systemic herbicide that can be used to control variable-leaf milfoil. Triclopyr mimics a member of the plant hormone group known as auxins, which are important in regulating the growth of dicot plants. An overdose of auxins causes the plant to lose control over its own growth and eventually die. As a systemic herbicide, triclopyr kills the entire plant and may therefore achieve control of target species over multiple seasons.

Triclopyr has achieved good control of variable-leaf milfoil in some northeastern ponds (Getsinger et al. 2003, Netherland and Glomski 2008). However, this level of control may require a long period of treatment at high concentrations, which can severely reduce cost-effectiveness and somewhat increase the potential impacts to non-target organisms. Triclopyr treatments are comparatively expensive, as much as $1,000 per acre.

2,4-D – Systemic Herbicide: The systemic herbicide known as 2,4-D (trade name Navigate) is dicot-selective and frequently effective in controlling growths of variable-leaf milfoil over multiple seasons. As with triclopyr, 2,4-D mimics a member of the plant hormone group known as auxins, which are important in regulating the growth of dicot plants. An overdose of auxins causes the plant to lose control over its own growth and eventually die. The primary advantage of 2,4-D is that it has been in use for a long time

and is available at a lower cost than other systemic herbicides. Applications of 2,4-D are typically in the range of $500/acre.

The primary drawback to this herbicide is that it is not effective against fanwort, the most extensive invasive species in the pond. Therefore, it is not recommended at this time.

Glyphosate – Systemic Herbicide: Emergent plant growths of exotic species of (purple loosestrife, common reed, and yellow flag iris) in Whitman's Pond could potentially be controlled with the herbicide glyphosate (trade name Rodeo) on a selective basis, if desired. Glyphosate is fast-acting for a systemic herbicide. It works by inhibiting production of key amino acids in plants and is only selective in that it is not effective on submersed vegetation. However, non-target emergent (e.g. cattail, sedges, rushes) or upland plants may be damaged or killed if they are exposed to glyphosate.

Growth of common reed is somewhat extensive on the western end of the West Cove but occurs in relatively small beds in the Main Basin and South Cove of Whitman's Pond. If desired, common reed could be treated with glyphosate. However, given the extensive rhizome network present in common reed beds, it is likely that multiple applications would be necessary to ensure dieback of all roots. Leaving viable rhizomes in place could result in complete regrowth of common reed within a few growing seasons. Given the limited size of common reed beds in the Main Basin and South Cove, as well as the nearly inaccessible beds at the western end of the West Cove, glyphosate treatments are unlikely to provide much benefit for the expense. Therefore, use of glyphosate to control common reed is not currently recommended.

Purple loosestrife infestation at Whitman's Pond, particularly in the South Cove, appears to be advanced and dense enough that biological control may be quite effective as a primary control (See section 5.1.2). It may be possible to achieve faster control through an integrated program of appropriately timed glyphosate applications and biological control, but this would reduce the cost-effectiveness of the control program. Purple loosestrife does not currently appear to inhibit recreation, water supply, or habitat for most wildlife or fish. Therefore, incurring additional expense to speed the control process would have minimal benefit and does not appear to be justified at this time.

Glyphosate treatment is not necessary for water lily control but could be used in tandem with hydroraking to improve the efficiency of a hydroraking operation. Application of glyphosate to floating water lily leaves would weaken the plants and make it easier to remove entire plants with a hydrorake.

Glyphosate control of yellow flag iris is not currently necessary, given the diffuse nature of the infestation. Isolated patches of yellow flag iris may be better controlled through routine monitoring combined with hand harvesting (see Section 5.1.5). Costs for such a minimal program to be implemented would be on the order of $1,500 per year with reporting.

5.1.2 Biological Control – Recommended only for Purple Loosestrife

Biological control involves the introduction of a predator, herbivore, parasite or other type of agent that inhibits the growth or reproduction of the target species. Biological controls can be useful in helping to reduce the size of active infestations but rarely result in eradication of a target species. Furthermore, they usually do not work as rapidly as chemical or mechanical management techniques. Depending on the size of the infestation and the nature of the biological organism used for control, it may take five to seven years before a significant level of control is observed.

Loosestrife Beetles

The only currently feasible biological control approach in Whitman's Pond is the culture and targeted release of loosestrife beetles (*Galerucella spp.*) for the control of purple loosestrife. Loosestrife beetles tend to stay within a small territory, especially when beetle density is low, which makes natural dispersal of populations very slow (NCERA-125, 2008). Consequently, they work best as a control method where contiguous stands of purple loosestrife occur.

Although the adults are the most visible life stage, it is actually the larvae that play the biggest role in control of purple loosestrife plants. Damage from adults is mostly limited to superficial leaf damage, which is unlikely to weaken the plant substantially. However, larvae burrow deep into stems and can therefore kill back entire shoots. Therefore, it may take several years for loosestrife beetle populations to sufficiently reproduce and grow to a density that makes a measurable difference in purple loosestrife cover.

Additionally, loosestrife beetles are unlikely to eradicate purple loosestrife infestations. This highlights one of the primary drawbacks of biological control using specialist herbivores, namely that a host population of the undesirable plant must be maintained in order to prevent the herbivore population from collapsing.

Loosestrife beetles are a highly selective control method because they are specialized herbivores on purple loosestrife. Therefore, the impact to non-target organisms is anticipated to be negligible.

Adult loosestrife beetles can be obtained (with a permit) at a cost of $275 to $300 for 1,000 beetles. Because adult beetles do less damage to purple loosestrife plants than larvae, releasing more adults at the beginning of the program does not necessarily imply faster control. The adult beetles must have a plentiful food source to encourage successful reproduction. An initial release of up to a few thousand adults in a handful of select locations is recommended. Additional beetles may either be purchased in future years or reared by volunteers on container-grown purple loosestrife plants. Monitoring of purple loosestrife damage and the loosestrife beetle population are the best way to determine whether additional beetles need to be released in subsequent years. However, as a general guideline, repeated releases of adult loosestrife beetles should be planned for the first two to three years of the control program.

It is recommended that release of adult beetles be limited to significant contiguous infestations, such as the area around the inlet of Old Swamp River in the South Cove. Isolated purple loosestrife infestations along the shoreline or in the emergent wetlands of the West Cove may be best controlled by manual removal, which can be conducted by trained volunteers.

Other Controls Assessed but not Currently Recommended

The milfoil beetle (*Euhrychiopsis lecontei*) is a native species that originally infested indigenous northern milfoil (*Myriophyllum sibiricum)*. However, exotic Eurasian milfoil (*Myriophyllum spicatum*) also serves as an acceptable host for milfoil beetles and may sometimes be controlled by introducing milfoil beetles to an infested pond. The larvae of this beetle burrow into the stems of the Eurasian milfoil plant, consuming the plant tissue within the stem and ultimately causing the plant to collapse. The best results are usually achieved in lakes with dense, monotypic stands of Eurasian milfoil and several years are typically required to measure a positive effect. Whitman's Pond is not currently known to host Eurasian milfoil. Therefore, the water milfoil beetle approach would not be appropriate at this time.

Biological controls for other plant species are almost unknown. An herbivorous fish (*Ctenopharyngodon idella*, the grass carp) has been used for general macrophyte control on an experimental basis in smaller lakes in Connecticut, New York, and Virginia. This species does not appear to show a preference for any

one plant species, resulting in large areas devoid of aquatic plants and, ultimately, the need to replant the treated pond with native plants (a costly proposition). Stocking of grass carp is not currently legal in Massachusetts and can therefore not be recommended as an appropriate approach for Whitman's Pond.

5.1.3 Bottom Sealing – Recommended for Use over Limited Shorline Areas

Bottom sealing involves the use of negatively buoyant materials, usually in sheet form, to create benthic barriers, i.e., barriers that limit plant growth by obscuring light penetration, physically obstructing growth, and encouraging chemical reactions that are unfavorable to plants.

Benthic barriers may provide control of exotic milfoils, fanwort and other nuisance growth on a localized basis. They are best used in heavily used recreational areas near shore and in the vicinity of docks or other shoreline structures, where growth of aquatic plants is not desirable.

Barrier materials have been commercially available for decades and a variety of solid and porous are available. However, deployment and maintenance of benthic barriers continues to be difficult and this limits their utility over the full range of weed bed sizes.

Plants under the barrier will usually die about a month following installation. Once plants have been killed, barriers of sufficient tensile strength may be redeployed to a new location if desired. However, keeping barriers in place for the entire season or multiple seasons is usually desirable because it prevents colonization by nuisance species.

The ability of vegetative fragments to colonize porous benthic barriers such as fiberglass screening has made them less useful for combating infestations, as sheets must be removed and cleaned regularly, often yearly. Solid barriers are more effective in killing the whole plant and preventing colonization of the area from new seeds or fragments. However, the gas released during decomposition in the sediments below can cause solid barriers to billow, necessitating the use of anchors or vents that can reduce the lifespan of the barrier itself. A drawback of all benthic barriers is that they are only effective as long as they are in place. Colonization from adjacent plant beds can occur quickly once the barrier has been removed.

One significant drawback to benthic barriers is that they are non-selective, which means that all plants in the treatment area are killed, including desirable native plants. By smothering bottom sediments, barriers may also impact the benthic macroinvertebrate community within the treatment area. In addition to causing direct mortality to native mussels and other macroinvertebrate organisms, barriers may locally reduce food sources for bottom-feeding fish.

Cost and labor are the main factors limiting the use of benthic barriers and would be prime deterrents for large-scale use in Whitman's Pond. The cost per installed square foot is on the order of $1.50 to $2.00, leading to an expense approaching $90,000 per acre. Bulk purchase and use of volunteer labor can greatly decrease costs, but use over large areas of nuisance vegetation is highly unlikely.

Benthic barriers could be useful by the Town or private landowners to address nuisance plant growth along small shoreline areas, where deployment and any subsequent maintenance would be relatively simple. A small installation immediately offshore from public access locations may be worth considering in tandem with other management approaches.

5.1.4 Hydroraking and Rotovation – Recommended for Limited Control of Nuisance Water Lilies

Hydroraking uses a backhoe-like machine mounted on a barge to remove plants directly from pond sediments. Depending on the attachment used, plants are scooped, scraped, or raked from the bottom and deposited on shore for disposal. Rotovation is essentially underwater rototilling of pond sediments. Rotating blades cut through roots, shoots, and tubers, dislodging and expelling them from their growing locations. Some operations are also outfitted to collect some or most of the rotovated plant materials.

Both hydroraking and rotovation are most useful for local control of water lilies and other plants with large rhizomes or tubers, as these methods can physically remove or destroy the bulky portions of the plant.

Hydroraking would only be recommended in areas where water lilies are considered to be problematic and other options for treatment are not preferred. It is not recommended for control of vegetatively reproducing species such as variable-leaf milfoil and fanwort.

Although hydroraking would be expected to target water lilies, the rake attachments are coarse tools that would be expected to capture significant bycatch of non-target aquatic plants and animals (primarily invertebrates)..

Costs to perform hydroraking vary depending upon a number of factors, including plant density, distance of the target beds from shore, and size of the area to be managed but an approximate cost of $6,000 to more than $12,000 per acre should be anticipated, if performed by a contractor.

A hydrorake could also be purchased by the Town, although ongoing maintenance and operation costs should be anticipated in addition to capital costs for initial purchase. Capital costs alone may exceed $100,000.

The Town currently possesses an Order of Conditions to conduct hydroraking operations (with prior approval from the Conservation Commission). However, depending on when hydroraking is additional costs may be required to to extend or reapply for a permit. Additional local permitting costs to obtain an Order of Conditions from the Conservation Commission would also apply (typically $4,000 to $5,000, if filed as a standalone NOI). Hydroraking would need to be repeated periodically, perhaps every three to five years to maintain the desired conditions.

5.1.5 Macrophyte Harvesting – Recommended for Small Scale Control Only

Macrophyte harvesting actually covers a wide range of techniques, including mechanical harvesting, diver assisted suction harvesting (DASH) and hand harvesting. Each of these involves direct removal of plant biomass from the water column but the appropriate circumstances and level of control achieved by each is likely to be much different.

Hand Harvesting

The simplest form of harvesting is hand pulling of selected plants. Depending on the depth of the water at the targeted site, hand pulling may involve wading, raking, snorkeling, or SCUBA diving. Hand harvesting often involves collection of pulled plants and fragments in a mesh bag or container that allows for transport and disposal of the vegetation. In deeper water, frequent trips to the surface are necessary to dispose of full bags. The intensive nature of this work limits its application to small areas, typically much less than one acre in size. Hand pulling can directly confirm removal of entire individual plants, typically resulting in longer control of plant growth in targeted areas.

In Whitman's Pond, hand harvesting would be most appropriate for the control of isolated purple loosestrife and yellow flag iris plants along the shoreline. It could also be used as a follow-up to other methods for control of aquatic plants, particularly where treatment of dense or extensive beds was not completely effective.

Hand harvesting is highly selective once the individuals pulling plants are trained to identify the target species. Some non-target organisms may be disturbed during harvesting but this disturbance is generally small-scale and consistent with impacts from normal recreational activities.

With proper training, volunteers could perform most of the shallow-water hand harvesting efforts, significantly reducing the cost of this option. Deeper beds are a much more challenging prospect and best harvested by trained professional divers. Diver harvesting typically costs $1,500 to $2,000 per day and the rate of progress depends both on plant density and diver expertise. However, it would be unusual for a team of two divers to achieve more than ¼ acre of harvesting per day, even in a lightly populated plant bed.

DASH

DASH is similar to simple diver harvesting but is more efficient because entire plants are fed into a suction hose and lifted to a collection vessel at the surface, thereby significantly reducing the time it takes for the diver to handle and return plants to the surface. DASH may proceed at a rate of ¼ acre or more per day depending on weed bed density. DASH costs range from $2,000 to $5,000 per acre depending on the density of plants and visibility in the water.

In Whitman's Pond, DASH would be most appropriate as a follow-up to other methods for control of aquatic plants, particularly where treatment of dense or extensive beds was not completely effective. For example, DASH could be a useful technique for removing remnant variable-leaf milfoil beds following a Sonar (fluridone) treatment.

As with hand harvesting, DASH is highly selective. Some non-target organisms may be disturbed during operations but this disturbance is limited given the relatively small area that would be affected at any given time.

DASH could also potentially be used as a primary control method for fanwort and variable-leaf milfoil. However, the costs and timeline for implementing such a program would be significant. Assuming average progress of ¼ acre per day, removal of all 76 acres of fanwort would require more than 300 working days of DASH. This implies that work would need to be done over multiple seasons and/or using multiple dive teams for a cost of approximately $150,000 to $380,000. Given the high density of fanwort and mucky sediments characteristic of the South Cove, West Cove, and portions of the Main Basin, costs are likely to be closer to the upper end of this range for a single pond-wide DASH campaign. Additionally, follow-up diver harvesting or DASH operations would be recommended as soon as possible to remove any regrowth from missed roots or plants before they have a chance to fragment and establish new beds. Although this effort would likely proceed much faster than the initial DASH campaign, significant (i.e., at least $20,000 and possibly much more) additional costs should be anticipated for the follow-up effort as well.

Mechanical Harvesting

Mechanical harvesting, which involves cutting and pulling aquatic plants from a specially-equipped watercraft, is the most effective harvesting method in the short term. However, as mechanical harvesting simply sets plants back for the season (akin to mowing a lawn), its use should be reserved for scenarios where there is an immediate but temporary need for widespread reduction of nuisance plant cover. Such a scenario could involve clearing around a water intake to prevent clogging during pumping.

Unlike other forms of harvesting, mechanical harvesting is non-selective. Significant bycatch of non-target aquatic plants and animals should be anticipated if this management option is selected.

Mechanical harvesting is not currently a recommended management option for Whitman's Pond because it is relatively expensive, typically results in only single season control, and may not be physically feasible near the Middle Street boat ramp in the Main Basin or in many areas of the South Cove and West Cove (the harvester cannot operate in very shallow water). Furthermore, the dominant plants of concern are

variable-leaf milfoil and fanwort, which both spread through vegetative fragmentation. If other techniques are used prior to or concurrent with mechanical harvesting, implementing this method would only encourage re-colonization of the targeted management areas by variable-leaf milfoil and fanwort.

5.1.6 Resident Waterfowl Control

Large resident Canada Goose populations have become established in eastern Massachusetts over the last 50 years, where hunting restrictions and feeding by the public have reduced pressures on this species. ESS observed Canada Goose flocks of 20 to 40 birds using the pond and adjacent shorelines over multiple visits. Based on our observations, the most utilized areas during the daytime hours appeared to be along the western (Middle Street) and northeastern shorelines of the Main Basin.

Canada Geese cross Middle Street in front of oncoming traffic

High densities of resident Canada Goose may contribute to excess nutrient and bacteria loading at Whitman's Pond. Geese crossing Middle Street cause numerous traffic backups. Additionally, resident geese may become aggressive toward children and adults that approach too closely. Management of the resident Canada Goose population at Whitman's Pond is recommended to help reduce the incidence of these problems.

Management of the resident Canada Goose population is most likely to be accomplished if multiple active and passive control options are implemented. Recommended actions include modifying lawn care practices near the pond and maintaining fences or vegetated buffers around the pond shoreline. Additional options, such as egg addling, use of chemical repellents, harassment, expanding public education and installation of decoys could also be of some use but are generally less cost-effective or controversial methods.

Raising the cutting height on lawnmowers is the simplest way to reduce grazing by Canada Goose in lawns that are accessible from the pond. Geese find taller grass to be less palatable. This method would also have the added benefit of reducing the time and money spent on lawn maintenance by residents and the Town.

Installation of fencing or re-landscaping the immediate shoreline to incorporate a buffer of shrubs and larger herbaceous plants would help make these areas less attractive to grazing geese. When geese molt over the summer, they are unable to fly and loathe to pass between obstacles that obscure their vision of potential predators. A wide variety of fencing materials could be used for this purpose, as long as they extend the entire perimeter of the open shoreline transition area and are at least 30 inches tall. An unlocked gate may be an effective way to allow public access at the boat launch while preventing Canada Geese from grazing along the shoreline or crossing Middle Street.

The cost of vegetative buffers or barrier fencing is likely to range from $4.00 to $15.00 per linear foot for materials and construction, depending upon the materials used. Additional design and permitting fees (typically $3,000 to $5,000) would also be required.

5.2 Recommended Long-term Options

Long-term management options include those that either require significant planning or have an effective period greater than five years. Some long-term management options (e.g., water level control) may involve short-term actions implemented repeatedly to achieve long-term management goals. However, only minimal cost or effort is required each time the option is implemented.

5.2.1 Water Level Control (Drawdown)

Drawdown involves lowering the water level of a lake to expose shallow bottom sediments and associated plants (both native and non-native) to drying and/or freezing. It is most effective against species that reproduce mainly by vegetative means, including fanwort and variable-leaf milfoil. Drawdown is less effective on species that reproduce primarily by seed or turions (winter-hardy buds), such as curly-leaf pondweed and many native species.

Although drawdown can be conducted at any time, the interaction of drying and freezing that occurs with winter drawdown is usually most effective. Environmental restrictions and recreational uses also limit the appropriate window for drawdown to the winter period. In coastal Massachusetts, winters are variable in their intensity and the ideal winter condition of very cold weather with limited snow cover (which would otherwise insulate the plants) is not likely to be achieved more frequently than every other year. However, even if ideal macrophyte control conditions are not reached during a given drawdown cycle, some control may still be achieved under marginal conditions. Furthermore, a program that is repeated as necessary (often on a two to three-year basis) is likely to be successful over the long-term. Therefore, rather than trying to match the perfect weather conditions up with a single lake drawdown, water level control should be thought of as an ongoing, long-term approach.

"Ice rip" is a drawdown technique that focuses on physical removal of rooted aquatic plants by managing ice cover to literally "rip" the plants, including roots, from shallow areas. This technique is questionable in its overall effectiveness and is not recommended for Whitman's Pond as variable-leaf milfoil and fanwort spread primarily by fragments (not roots) and it is unlikely to be more effective than a standard winter drawdown program. Additionally, the rapid induced fluctuation of water levels and ice cover may exacerbate shoreline or downstream erosion, suspend bottom sediments and associated nutrients that are lifted with the ice, negatively impact bottom-dwelling fauna, disrupt hibernating reptiles and amphibians along the margins of the pond, reduce the safety of winter recreation activities on the ice, or compromise the dam.

In order to effectively drawdown a lake, there must be an adjustable discharge structure that allows the water level to be safely controlled and maintained at the targeted level. The water level must be drawn down to a sufficient depth (typically 3 feet) and for a long enough period of time to allow bottom sediments to at least partially de-water and freeze. Aside from the practical feasibility of performing a drawdown, the potential impacts on winter recreation (primarily ice fishing and skating) should also be considered.

The first step in pursuing a drawdown program would be to verify dam ownership and drawdown capability. Any manipulation of the water level in Whitman's Pond would need to be approved by and coordinated with the dam owner and the operator of the town's water supply (Water and Sewer Division).

A drawdown feasibility study would first need to be developed that would identify potentially sensitive habitats or biota that may be present within the pond, its downstream waters, or within hydrologically connected wetlands. This study would also examine the feasibility of drawdown with regard to controlling hydraulics (related to the amount of water Whitman's Pond can hold, how much would be lost during the drawdown, and limitations concerning where the water goes downstream), flooding, and impacts to downstream and hydrologically connected wetland resources (e.g., drying) and would be used to establish a current baseline condition as well as to support permitting.

In addition, a Drawdown Operations Plan would need to be developed, inclusive of all hydrologic calculations, which will serve to guide dam operators on methods for managing the drawdown timing, the release rate, and the magnitude of drawdown. The Drawdown Operations Plan will also provide protocols for monitoring the system to ensure protection of biota within pond and associated waters while also achieving a better level of control on the targeted macrophyte species. Additionally, this plan should establish a protocol to ensure the pond is filled to a level sufficient to restore the required minimum flow through the fishway in time for alewife spawning season. Given the substantial amount of relevant data already collected under the current study, the costs for performing the drawdown feasibility study and preparing the Drawdown Operations Plan are likely to be on the order of $8,000.

Once this information has been determined and the Drawdown Operations Plan is developed, it will then be necessary to file a Notice of Intent application with the Conservation Commission. Chapter 91 permitting would also be required to modify the water level of a Great Pond. Assuming that a Drawdown Operations Plan is made available, filing a permit to conduct a drawdown at Whitman's Pond is likely to cost between $6,000 and $8,000 to prepare and file based upon the nature of the impacts and the supporting studies.

The outlet control structure at Whitman's Pond appears to be capable of releasing water for a significant pond drawdown, although this should be verified prior to moving forward with drawdown planning and permitting. Additionally, under certain circumstances, it may be desirable to draw down the South Cove by pumping water to Weymouth Great Pond using existing infrastructure. For the most effective exotic aquatic macrophyte control, a target drawdown depth of six feet would be ideal. This depth would expose a significant acreage of sediments on the shallow western end of the Main Basin and provide the best chance for sediment dewatering and desiccation during the winter months. Under this scenario, approximately 76 acres along the shoreline of the Main Basin and South Cove would be exposed (Figure 12). This scenario could make a significant impact on the exotic macrophyte beds in the Main Basin and South Cove, thereby potentially reducing or eliminating the need for extensive herbicide treatments in these areas.

Alternatively, should a reduced (e.g., four-foot) drawdown be implemented, 42 acres of sediment exposure could be expected (Figure 12). While this would still impact much of the South Cove, the area of potential control in the western portion of the Main Basin would be much more limited.

Despite the multiple advantages of drawdown, some negative impacts are possible. Drawdown reduces habitat volume for overwintering fish populations and amphibians may be sensitive to fluctuating water levels if it exposes them to dry or freezing conditions. Additionally, invertebrate species, particularly freshwater mussels, may be desiccated or frozen if drawdown occurs too rapidly. Lastly, drawdown may favor certain plant species over others, resulting in a shift in the plant community. However, since it tends to have a greater impact on rooted plants that spread primarily by fragmentation (e.g., fanwort and milfoils), this shift would be expected to be mostly positive. Plants that spread by seed or winter buds or are not rooted would be less impacted or even encouraged by drawdown. These include many of the desirable native species at Whitman's Pond (e.g., small bladderworts, native pondweeds, and water celery) as well as the exotic curly-leaf pondweed. A monitoring program would likely be required to ensure that drawdowns are protective of aquatic organisms and do not result in undesired impacts on the plant community, including spread of curly-leaf pondweed.

If a six-foot drawdown is not technically feasible, approved through the permitting process, or otherwise desired, drawdowns as small as three feet may still be worthwhile for providing some macrophyte control along shoreline areas and reducing the area that would need to be controlled through more costly measures, such as herbicide application.

If drawdown were successfully permitted, an annual monitoring program to track any impacts to aquatic resources would likely be required as a permit condition. A typical monitoring program will include monitoring of in-pond water quality, freshwater mussel populations, and hydrologically connected wetland plant communities, as well as availability of fish and wildlife habitat. In addition, it is recommended that plant cover in the pond be mapped on at least an annual basis so that the progress of the drawdown program can be documented and recommendations adjusted, as needed. Implementation of a typical drawdown monitoring program could be expected to cost approximately $7,000/year.

5.2.2 Dredging

Dredging works as a plant control technique when either a light limitation is imposed through increased water depth or when enough soft sediment is removed to reveal a less hospitable substrate for plant growth (e.g. hard bottom or other nutrient-poor substrate).

Conventional "dry" dredging requires pond drawdown

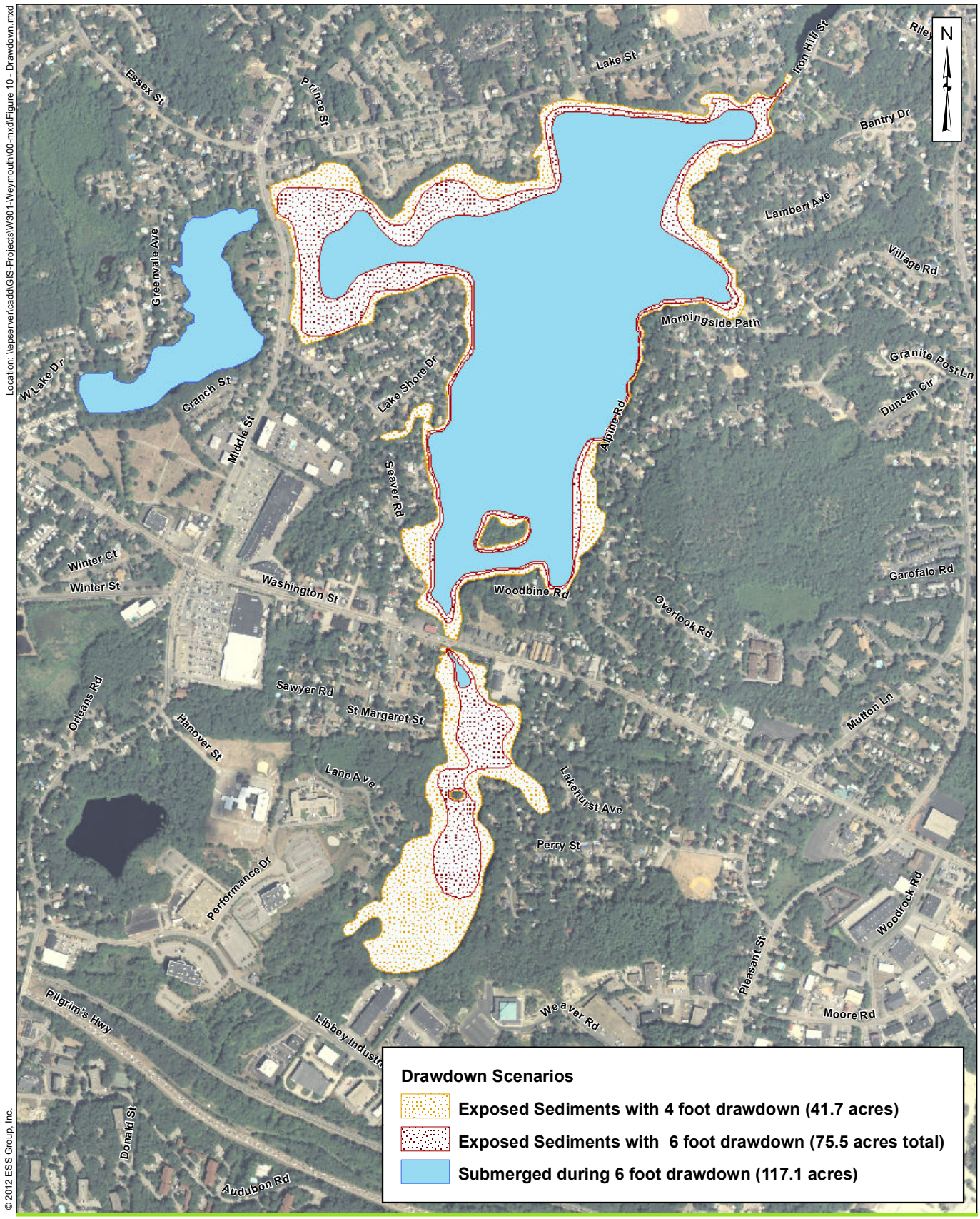
Light limitation through increased depth may be possible in Whitman's Pond, in part because the natural staining of the water provides some reduction in the amount of light penetrating to the pond bottom. Fanwort and variable-leaf milfoil were not observed to grow in waters deeper than approximately eight feet in Whitman's Pond. Therefore, dredging should target at least this depth for the reduction of exotic invasive plant growth. Dredging to greater depths would provide additional storage volume, increase the likelihood of success in controlling nuisance plant growth, and allow for benefits to be sustained longer. In areas where thick deposits of soft sediments have accumulated (greater than 12 feet in some locations), deeper dredging would have the added benefit of removing a significant nutrient source, thereby reinforcing the control of fanwort and variable-leaf milfoil beds.

Because dredging involves removing pond sediments, it is considered a non-selective management technique that will affect non-target plant species and some animals (primarily invertebrates) living in the immediate dredged area.

Dredging in Whitman's Pond could be an effective long-term control technique for nuisance aquatic plants. The challenges of dredging projects are not unreasonable and the potential long-term benefits can be significant.

The portion of Whitman's Pond where dredging may be most beneficial is the South Cove and the following discussion will focus on this basin. Dredging could also be used as a vegetation management action in portions of the Main Basin or West Cove. However, in these basins, dredging costs would likely be higher while achieving a narrower range of benefits.

If dredging were completed in the South Cove, the storage capacity of Whitman's Pond would be increased. While the change in storage would be expected to affect routing of flows through the system (by attenuating peak flows), it would not be anticipated to directly reduce the overall volume of water flowing out of the pond into Herring Brook.



VEGETATION ACTION MANAGEMENT PLAN
Whitman's Pond, Weymouth, MA

1 in = 1,000 ft

0 500 1,000 Feet

Source: 1) NAIP Orthophotography, 2009
2) ESS Bathymetry Data

Four- and Six-foot Drawdown Scenarios

Figure 12

Geotubes used to dewater hydraulically dredged material

A key factor influencing the approach and costs for moving forward with a dredge program at Whitman's Pond is the ability to draw down the pond to allow for dredging within the drained basin to occur using conventional excavation equipment. Conventional (dry) dredging is most easily conducted during the winter months when cold temperatures allow for more efficient excavation and transport of fine sediments.

This approach would allow for sediment to be dewatered within the pond itself by pulling the sediment up to the shoreline edge and allowing water to drain back into the pond basin.

Some use of temporary cofferdams or in-basin diversion channels to route the flow of water around the work area may be necessary near major inputs of surface water, such as the Old Swamp River.

If conventional dredging is not determined to be feasible for Whitman's Pond due to equipment access issues or water supply management concerns, hydraulic (wet) dredging could be a viable alternative. Hydraulic dredging is generally more expensive per cubic yard than conventional dredging and it typically requires a larger and more sophisticated containment area to dewater the sediment as it is removed from the pond. Alternatively, advanced dewatering techniques such as the use of Geotubes (geotextile fabric for dewatering) or a belt-filter press machine could be used. However, these approaches may add costs over traditional dewatering containment. All of these options require land adjacent to the pond to be made available for the dewatering process.

The Washington Street pumping station lot might provide adequate space for the use of a belt-filter press machine but a larger area would be required for Geotubes (one acre or more) or a standard dewatering basin (at least two acres). Pumping South Cove sediments into a small hopper barge and using the Middle Street public access area for dewatering could potentially provide an alternative option but would significantly slow down the operation, further increasing the cost of the project.

Environmental permitting for dredging projects is fairly complex and may require up to a year to secure all necessary approvals. Federal (US Army Corps of Engineers Section 404), state (MEPA Certificate, Section 401 Water Quality Certification, and Chapter 91 Waterways Permit) and local permits (Notice of Intent filed for Order of Conditions from the Conservation Commission) would be required.

Costs

The amount of material to be removed and the type of disposal or reuse will have a significant impact on the cost of dredging. With an estimated soft sediment volume of approximately 275,000 cubic yards, the cost of a dry or hydraulic dredging project for the entire South Cove would likely run between $4.5 million and $6 million (including permitting and design) for removal of all of the soft sediments. Costs could increase if sediment cannot be reused or disposed of in the immediate vicinity of the pond.

A more realistically scaled project designed to deepen the northern portion of the South Cove, which is adjacent to the existing water intake and appears to have the cleanest sediments, might reasonably yield a dredge volume of 75,000 to 100,000 cubic yards. Costs to dry dredge this volume of material would likely range between $1.5 million and $2.5 million with permitting and design costs likely to add an additional $120,000 to this total.

If dry dredging is not determined to be desirable or feasible in the South Cove, hydraulic dredging for a similar scale project would range between $1.9 million and $4.5 million, depending on the dewatering method selected,with permitting and design adding an additional $120,000 in costs.

Chemical content of the material to be dredged is an important consideration in determining the feasibility of reuse or disposal. Disposal costs could be much reduced if the material removed from the pond is clean enough for beneficial use as a soil amendment. Clean material could potentially have value to a local landscape supply, golf course, or other business which would reduce disposal costs or even help offset the cost of the project. However, material that is not suitable for beneficial use would need to either be amended with clean material to dilute the concentrations to suitable levels or trucked offsite for disposal. Either of these options would increase the cost of the project.

MassDEP will make a final determination on suitable reuse options for dredged material as part of the permitting process. However, based on the sediment sampling results obtained as part of this study (Appendix D), sediment from the northern portion of the South Cove is the most likely to be suitable for upland reuse either as is or after minimal amendment with clean material. Sediments appear to contain progressively greater concentrations of contaminants (mainly metals) as one moves south in the South Cove, and which suggests that more significant amendment or offsite disposal could be required.

If dredging is pursued as a desirable long-term option, the next steps would be as follows:

1. Assessment of a specific scope and extent of dredge program, including possible funding options.
2. Additional chemical and physical analysis of the sediments in areas targeted for dredging – level of effort is based on the volume of material targeted to be dredged.
3. Development of an engineering design for submission to permitting authorities.
4. Initiation of the permitting process including an Environmental Notification Form filing for MEPA (Massachusetts Environmental Policy Act) review, filing a local Notice of Intent under the Wetlands Protection Act, filing for a Section 401 Water Quality Certificate and Chapter 91 Permit from MassDEP, and seeking a U.S. Army Corps of Engineers Section 404 Permit for dredging.

These four activities might be expected to cost approximately $80,000 for Whitman's Pond given the work already completed as part of this study, but are essential if dredging is to be pursued as a management option. Additional design costs would include final engineering design following the permitting process (incorporating any accepted changes resulting form these reviews) along with the development of a bid specification package for the project.

5.2.3 Control Nutrient and Sediment Loading

Nutrient loading analysis was beyond the scope of this study. However, based on data collected in previous studies, the current condition of Whitman's Pond, and the nature of land use in the watershed, it will be important to continue to reduce nutrient and sediment loading to the pond. Effectively enforced stormwater by-laws in Weymouth can help prevent further loading by eliminating illicit discharges and requiring new developments and redevelopments to incorporate Low-impact Development (LID) storm water techniques. Additionally, good housekeeping of existing features, such as frequent street sweeping, regular catch basin cleaning, and maintenance of the sedimentation nutrient uptake pond (SNUP) on the Old Swamp River, can help prevent nutrient and sediment loading to Whitman's Pond. Proper operation and maintenance of these features is key to ensuring that they continue to function as designed.

Some existing stormwater features may need significant upgrades to prevent sediment and nutrients from entering.Whitman's Pond and further fueling eutrophication of the pond. This is particularly evident in the West Cove, where stormwater outfalls, such as the Cynthia Circle outfall, are numerous.

Control of nutrient and sediment loading would not be expected to have a negative impact on non-target species.

5.2.4 Other Recommended Approaches

Public Education and Outreach

Public education and outreach are will raise awareness of issues at Whitman's Pond and encourage public involvement in its protection and management as a community resource, particularly with regard to prevention of future problems. Education and outreach may take many forms. These may include postings at the Middle Street kiosk, mailings, school programs, booths at Town-sponsored events, and website postings, to name a few.

Organized volunteer programs also provide an opportunity for members of the public to take a more active role in protecting Whitman's Pond. Examples of such a volunteer program could include a Town-sponsored boat monitor program at the Middle Street boat launch. Alternatively, the Massachusetts Weed Watchers program, sponsored by the Department of Conservation and Recreation Lakes and Ponds Program, provides training and technical assistance to volunteer groups interested in monitoring and reporting exotic species. Either of these programs would be helpful for preventing establishment of new exotic species in Whitman's Pond.

Boating Channels

Boating channels are simply areas where aquatic plant growth is controlled so that boats may pass from public access areas to open water without crossing through invasive weed beds. They serve the dual purpose of providing easier access to open water as well as reducing propeller-induced fragmentation of aquatic invasive plants. This is particularly useful for preventing re-colonization of pond areas where aquatic invasive plant growth has been successfully controlled. Unless a municipal ordinance is adopted and enforced, success of this approach relies on a high level of voluntary compliance. Therefore, to realize maximum benefit, boating channels should be clearly advertised and well-maintained. For example, rope floats or other buoys may be used to delineate channels during the growing season. The boating channels themselves may be maintained by implementing one or more of the previously presented management options.

5.3 Options Assessed but not Currently Recommended

5.3.1 Plant Competition

Plant competition techniques seek to establish desirable native species through seeding and planting disturbed or bare areas before they can be colonized by invasives. The overall goal of the technique is to maximize spatial resource use by desirable species to keep out undesirable invasive species (Wagner, 2004).

The primary advantages of this approach are that it uses natural processes to control aquatic invasives, may be self-perpetuating after an initial establishment period of several years and can be easily integrated with other approaches.

However, plant competition techniques are still experimental, which makes the long-term effectiveness of this approach uncertain. In some habitats, native species may not grow quickly or densely enough to prevent successful re-invasion by exotics. Even if these species are able to suppress re-growth of exotic invasive weed beds, periodic natural disturbances within the plant community may provide numerous opportunities for invasive species to colonize. Therefore, significant ongoing effort to replant with supplemental native plantings could be required (Wagner, 2004).

A local Order of Conditions from the Weymouth Conservation Commission would be required to implement a plant competition approach in Whitman's Pond. Not including permitting, costs for

implementing this approach would vary depending on the species used and size of the area being planted, but estimates of more than $5,000 per acre for the initial planting effort would not be unexpected.

Plant competition is not currently recommended as part of the Vegetation Management Plan in Whitman's Pond because of its high initial cost, experimental status, and the potential for multiple years of ongoing material and labor costs to supplement native plant community. Additionally, as evident from the plant surveys, Whitman's Pond still appears to host a diverse native seed bank and significant regrowth of desirable native species may occur on its own once exotic invasive species are brought under control.

5.3.2 Dilution, Flushing, and Hypolimnetic Withdrawal

Dilution and flushing involve increasing the flow rate so as to dilute or remove concentrations of nutrients or other pollutants in the lake. Similarly, hyplimnetic withdrawal involves removing oxygen-depleted waters from the hypolimnion to encourage mixing of oxygen-rich waters into this zone (with subsequent sequestration of phosphorus. Each of these options requires an appropriate outlet structure or sufficiently sized withdrawal device and must take into account the potential downstream impacts of increased flow and "flushing" of nutrients.

Due to the relatively large volume of Whitman's Pond, large inputs of clean water would need to be continually supplied to effectively dilute the concentration of phosphorus in the water column. Such a project would require significant planning and be costly to implement. Additionally, these approaches would not directly address the nutrients in pond sediments that are responsible for sustaining excessive aquatic plant growth in Whitman's Pond. Therefore, dilution, flushing, and hypolimnetic withdrawal are not currently recommended as part of the Vegetation Management Action Plan.

5.3.3 Shading Dye

Dyes are used to limit light penetration and therefore restrict the depth at which rooted plants can grow. In essence, they mimic the effect of light inhibition that might be expected during periods of high turbidity or prolonged ice and snow cover. Natural periods of low light are an important variable in determining plant composition and abundance, and use of dyes can produce similar effects. They are only selective in the sense that they favor species tolerant of low light or with sufficient food reserves to support an extended growth period (during which time the plant could reach the euphotic zone [area of the pond with sufficient light to sustain plant growth]). Dyes tend to reduce the maximum depth of plant growth, but are relatively ineffective in shallow water (less than 6 ft or 1.8 m deep). Dyes are unlikely to make a significant difference in plant growth within the shallower basins of Whitman's Pond and the high concentration would be difficult to maintain in the Main Basin, given its large volume. Therefore, the use of shading dye is not currently recommended.

5.3.4 Nutrient Inactivation

Nutrient inactivation typically targets dissolved phosphorus (the form most readily available to plants and algae) and involves the addition of alum (aluminum sulfate), iron(III) chloride or similar aluminum-based compounds that bind to this phosphorus to allow it to settle into the pond sediments. In its simplest form, nutrient inactivation is conducted by applying alum directly to the pond as a single dose. More sophisticated nutrient inactivation programs involve proportional injection of alum into stormwater sources or tributaries so that phosphorous is inactivated before it even enters the pond.

Compounds such as alum have some demonstrated effect on internal nutrient cycling but must be expertly applied and buffered to be effective while avoiding large pH swings and consequent collateral damage to sensitive organisms, such as fish and native mussels.

One new product that does not impact pH and appears to be essentially non-toxic consists of a blend of the rare metal lanthanum with bentonite clay (trade name Phoslock). This product is now registered for use in much of the United States but must be applied by a professional. The price for nutrient inactivation

Limno-barriers help confine treatment to small areas

with the lanthanum/bentonite mixture is quite high compared to traditional buffered alum and the additional benefits appear to be minimal.

Nutrient inactivation is typically used to control algae blooms and improve water clarity. It is an expensive option and one that is unlikely to result in a long-term reduction in growth of invasive aquatic macrophytes at Whitman's Pond. Therefore, this is not currently recommended as part of the Vegetation Management Action Plan.

5.3.5 Aeration and/or Destratification

Aeration and/or destratification (or circulation) is used to treat problems with high algal growth and low oxygen concentrations that may occur in smaller ponds. Air diffusers, aerating fountains, and water pumps are typical types of equipment that may be installed to increase circulation in a pond. The cost of purchasing, installing, and maintaining pond circulation equipment becomes substantial as pond size increases. Likewise, the effectiveness of the equipment tends to decline with pond size as it is difficult to achieve sufficient circulation in large ponds.

Aeration is not currently recommended for the Main Basin or West Cove of Whitman's Pond, primarily because sedimentation and excessive aquatic plant growth (rather than planktonic algal growth) are the targets for restoration of that portion of the pond. In the South Cove, where algae blooms have previously been problematic, aeration could potentially help control these blooms once macrophyte growth has been controlled. However, whether aeration may potentially be useful in the future depends on the success of primary methods used to manage the pond going forward. For example, should dredging be pursued in the South Cove, the removal of sediments and increase in water depth and volume could also have the benefit of preventing future algae blooms

The Main Basin is the only portion of the pond currently deep enough to be fully stratified during the growing season. However, algae blooms do not appear to be a significant problem in the Main Basin at this time. Additionally, aeration has been implemented on a limited basis in the past with little noticeable impact on water quality or plant growth. Therefore, aeration and destratification are not likely to be a useful approach at Whitman's Pond.

5.3.6 Novel Approaches

A number of novel approaches to aquatic vegetation management are available on the market, including bacterial formulations, barley straw, solar-powered aeration systems, floating islands, and many others. While some of these approaches have had success (anecdotal or data-supported) in addressing pond management issues, we are not aware of any not already addressed in previous sections that would currently be recommended to control the problems specific to Whitman's Pond.

One novel approach that ESS has successfully permitted elsewhere in Massachusetts is the use of limno-barriers. Limno-barriers are essentially floating curtains that extend from the water surface to the pond bottom. They can be used to isolate and treat small or pioneer infestations of aquatic nuisance species with registered herbicides or alternative methods (e.g., hydrogen peroxide) while minimizing the impact to surrounding waters and organisms.

Limno-barriers may be joined together in a closed loop to form treatment cells around isolated beds. Alternatively, they may be joined from one shoreline point to another to close off an entire cove. The alignment of the Whitman's Pond shoreline, particularly the Main Basin, implies that a significant length of limno-barrier material would need to be used to achieve the desired effect.

Limno-barriers do require an initial investment to purchase (or rent), which varies by the height and length of the material needed. Costs range from approximately $10 to $15/linear foot for 6- to 9-foot tall limno-barrier material, respectively. Additional costs for initial installation (approximately $2,000) should also be anticipated. Costs can be partially offset by savings on the amount of herbicide product needed for treatment as well as the fact that limno-barriers can be deployed by trained volunteers.

In Whitman's Pond, this approach could potentially be of value for maintaining the necessary dose of a liquid fluridone (Sonar) formulation in selected coves while minimizing exposure in non-targeted areas. It could also be useful for addressing re-growth of nuisance plant beds once the current infestations of fanwort and variable-leaf milfoil are brought under control.

5.3.7 No-action Alternative

The no-action alternative at Whitman's Pond would entail avoidance of all the management actions presented in the previous sections. If implemented, this option would allow exotic invasive plant species to continue to dominate while the pond fills with fine sediments and water quality stagnates or becomes increasingly poor. The pond would continue to serve some ecosystem function but the availability of open water habitats would likely decline. Similarly, the pond would likely continue to function as a recreational and water supply resource but with reduced area and volume, respectively. The West Cove and South Cove would be at highest risk for significant decline in pond services provided because of their smaller areas and lower average water depths (i.e., these are already marginal areas where continued exotic plant growth and sedimentation could result in conversion of remaining aquatic habitats to emergent wetlands).

Although this option does have the advantage of requiring no direct monetary costs, it may have a significant cost in the form of reduced aesthetic, recreational, water quality, water quantity, and ecological value. Some of this cost may be intangible; however, loss of recreational tourism, lowered waterfront property values, and the need to develop alternative water supply resources may result in real monetary costs to the Town and its residents. Therefore, the no-action alternative is not recommended.

6.0 MONITORING PROGRAM

A cost-effective monitoring program would provide continuous background data for the purpose of tracking the effectiveness of any future management practices at Whitman's Pond.

The key monitoring element associated with any vegetation management action program would be the mapping of aquatic plant species distribution, cover, and biovolume with particular focus on the distribution of exotic plant species. Biomonitoring of zooplankton (a key food for alewife and several other fish species) and sensitive macroinvertebrates (e.g., mussels) would also yield useful information for continued management of the pond and may be required under an Order of Conditions to implement certain management actions, particularly drawdown.

Water quality monitoring would also be useful to track in-pond conditions during the growing season each year. This could be used to identify any emerging negative trends in water quality before they become problematic as well as to document any improvements in water quality that may be realized through pond management actions or improvements in watershed management. Phosphorus and chlorophyll-*a* levels would be important in this regard, along with easily measured field parameters (pH, dissolved oxygen, temperature, specific conductance, turbidity, and clarity [Secchi depth]). Additionally, water quality monitoring would likely be required as a condition for herbicide treatment, particularly treatments that coincide with the recommended time-of-year restrictions for alewife spawning.

Evaluating water quality and plant coverage trends requires several years of continuous data, often with multiple sample dates in each year. Evaluation of management techniques would be more immediate, allowing comparisons between pre- and post-management periods. A program could be custom designed

to fit within an appropriate budget, but a cost of between $5,000 and $8,000 per year should be dedicated in order to include some level of water quality and plant community assessment along with a review of data by a qualified expert. Monitoring plant cover in the pond should be performed on at least an annual basis to track changes in beds of existing exotic species and identify any emerging infestations before they spread. Plant monitoring also allows evaluations of implemented management actions to be made and strategies adjusted, as necessary.

7.0 SUMMARY OF MANAGEMENT RECOMMENDATIONS AND CONCLUSIONS

The most critical management target identified through this study is the need to address invasive aquatic weed growth, particularly the extremely dense fanwort and variable-leaf milfoil present over most of the South Cove, West Cove and large areas of the Main Basin. These species grow in dense beds at biovolumes that inhibit recreational opportunities and reduce habitat for fish and wildlife that require open water or edge habitats. Curly-leaf pondweed does not yet appear to have established over large areas of the pond but does have the potential to be problematic. Many of the treatments recommended for fanwort and variable-leaf milfoil will also work to control this species. Purple loosestrife, common reed, and yellow flag iris, while problematic and undesirable in surrounding wetlands and shorelines, do not significantly impact water supply, in-pond recreational opportunities, or fish and wildlife habitat at this time. However, management of this species can be included for ecological or aesthetic reasons.

Excessive accumulation of fine sediments constitutes another management target. These fine sediments, which approach 18 feet thick in portions of the West Cove and South Cove, help to fuel the aquatic vegetation problems at Whitman's Pond and have the additional impact of reducing available water storage capacity for the Town water supply.

Water quality is another concern. Dissolved oxygen and phosphorus levels are problematic at times over large areas of the pond. Addressing internal and external sources of phosphorus and preventing the excessive growth and decay of aquatic plants will be necessary to ensure that water quality continues to be supportive of recreation, water supply, and fish and wildlife habitat needs.

In summary, ESS recommends that the following management options be considered for implementation at Whitman's Pond.

In the short term:

- Fluridone (systemic) and flumioxazin (contact) herbicides may be used for partial-lake or spot treatments of areas where quick control of the primary aquatic invasive plants (fanwort and variable-leaf milfoil) is desired. Fluridone may provide multi-season control while flumioxazin can only be expected to provide single-season control. Application of each herbicide may run up to $1,000 per acre.

  Fluridone could be used for initial control with follow-up spot treatments of flumioxazin on an annual basis. A three-year program involving fluridone treatment in one basin each year could be envisioned, beginning with a trial in the West Cove or South Cove to better establish the effects of treatment on dissolved oxygen levels and non-target species, then progressing to the Main Basin (as informed by monitoring results from previous treatment) in the second or third year. A budget of $20,000 to $35,000 per year for the first three years would likely be necessary, followed by $3,000 to $10,000 per year for annual follow-up treatments if herbicides are used as the primary method of control at the pond. Limno-barriers could be used to focus treatments in discrete areas, either for trials or as part of a regular, but contained, treatment program. Use of limno-barriers should be expected to have an additional cost of approximately $10 to $15 per linear foot of material required plus an initial installation fee of approximately $2,000.

- Hand harvesting, diver harvesting, and/or DASH can be used over small or isolated beds. These methods would be useful for controlling infestations along shorelines or in tandem with other methods to control infestations in deeper water. Costs vary depending on the density of beds and whether volunteers can do the work or professional divers are required. Professional diver harvesting and DASH should be expected to cost at least $2,000 to $5,000 per acre. This approach will be most cost-effective after the nuisance infestation is brought under control.
- Hydroraking can be used as a supplemental method to control tuberous or rhizomatous aquatic plants that cannot easily be hand harvested, such as water lilies. Costs for hydroraking can be high, ranging from $6,000 to more than $12,000 per acre. Control typically lasts three to five years, possibly longer.
- Resident waterfowl control is recommended to reduce the public health, public safety, and nutrient loading issues primarily associated with Canada Goose. The easiest and least expensive way to discourage resident waterfowl is to increase the mowing height of grassy areas near the pond shoreline. Installation of goose-proof fencing or natural landscaping could have a much larger, more immediate impact but would also incur design, permitting, and construction costs ($3,000 to $5,000 for design and permitting with and additional $4.00 to $15.00 per linear foot for materials and installation). Targeted educational materials (prepared for less than $3,000) distributed to local residents or in a kiosk at the pond could also be effective toward discouraging feeding as well as educating shoreline property owners about effective shoreline buffer controls.
- Benthic barriers may be useful for immediate control of infestations over very small areas, primarily around docks or public access areas. The cost of installed barriers is in the range of $2 per square foot, which translates into costs of about $5,000 for a typical 25 foot by 100 foot application.
- Biological control of purple loosestrife, primarily in the South Cove, could be pursued for relatively little cost. An initial introduction of loosestrife beetles can be used to control plants over the short-term. Long-term control could be achieved by continued re-introduction of beetles as needed or by hand harvesting loosestrife plants once the beetles have reduced plant density and extent. Costs to obtain adult beetles for release typically run $275 to $300 per 1,000 beetles.

In the long term:

- Careful design and implementation of a winter drawdown program could potentially be used to control aquatic weeds along the shoreline of the Main Basin and possibly in the South Cove for relatively little cost. The success of winter drawdown in the short term is highly variable, due to the vagaries of weather in any given winter. However, when viewed as a long-term management action, winter drawdown is often quite reliable.

  The first step in a drawdown program would be to complete a feasibility study and Drawdown Operations Plan (approximately $8,000) followed by permitting (an additional $7,000 to $8,000). If feasible, a six-foot drawdown would be anticipated to provide significant control of fanwort and variable-leaf milfoil in the Main Basin. Once the program has been permitted, a monitoring budget (typically $5,000 per year – less if combined with monitoring efforts associated with other management actions) will likely be required to assess the need for future drawdown and track any impacts to potentially sensitive resources.
- Dredging requires a substantial up-front investment but provides a chance to "start over" that no other single management option offers. At Whitman's Pond, dredging could be envisioned for multiple areas of the pond impacted by sedimentation and excessive aquatic plant growth. However, a partial dredging project in the South Cove appears to be the most cost-effective approach at this time.

Partial dredging in the South Cove would increase storage volume for the Town's water supply, while removing nutrients from the system and creating more open-water habitat.

A project designed to deepen the northern portion of the South Cove might reasonably yield a dredge volume of 75,000 to 100,000 cubic yards. Costs to dredge this volume of material would likely range between $1.5 million and $4.5 million with permitting and design costs likely to add an additional $120,000 to this total. Dredge costs are largely affected by approach (dry dredging usually costs less than hydraulic dredging), whether sediments can be beneficially re-used, and hauling distance.

Permitting of dredging is complex but can be successfully accomplished in as little as one year with proper planning and funding.

- Continuing to reduce watershed sources of all pollutants, but particularly nutrients and sediments, will be important for maintaining water quality and extending the life of any in-pond management activities. Much of the cost for this can be shifted to new developments and redevelopments through effective stormwater by-laws. Good housekeeping of existing features is also recommended.
- Ongoing education and outreach will raise awareness of issues at Whitman's Pond and encourage public involvement in its protection and management as a community resource. Active public involvement may be channeled into participation in a boat monitor program, Weed Watchers program, or a similar type of volunteer program.
- Sustained monitoring will also be a key part of ongoing management to track progress, prevent future infestations, and ensure preservation of the pond's recreational and ecological resources. A basic monitoring program can be established for approximately $5,000 to $8,000 per year. Monitoring may also be required as a permit condition for implementation of specific management actions and may entail additional costs. However, costs can often be reduced by merging common elements of separate monitoring programs.

The management of Whitman's Pond in a manner that is comprehensive and long-term will have significant initial costs. However, with clear goals, good data, proper planning and readiness to take advantage of funding opportunities as they arise, it can be accomplished. The work performed to date should be followed-up with real action as soon as possible to take advantage of the current data and momentum and ensure that progress continues.

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Appendix A

Quality Assurance Project Plan for the Whitman's Pond Vegetation Management Action Plan (Electronic Version Only)

Quality Assurance Project Plan

Whitman's Pond
Weymouth, Massachusetts

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ESS Project No. W301-000.02

Revised July 6, 2012

www.essgroup.com

QUALITY ASSURANCE PROJECT PLAN
Whitman's Pond
Weymouth, Massachusetts

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QUALITY ASSURANCE PROJECT PLAN (QAPP)

for the

Whitman's Pond Vegetation Management Action Plan

Revised July 6, 2012

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LIST OF ABBREVIATIONS

Abbreviation	Definition
CLM	Certified Lake Manager
cm	Centimeter
DGPS	Differential Global Positioning System
EIT	Engineer-in-Training
EPH	Extractable Petroleum Hydrocarbons
ESS	ESS Group, Inc.
g	Gram
L	Liter
MassDEP	Massachusetts Department of Environmental Protection
MDL	Method Detection Limit
mg	Milligram
mL	Milliliter
NA	Not Applicable
PAHs	Polycyclic Aromatic Hydrocarbons
PCBs	Polychlorinated Biphenyls
PE	Professional Engineer
PG	Professional Geologist
PWS	Professional Wetland Scientist
QAP	Quality Assurance Plan
QAPP	Quality Assurance Project Plan
QA/QC	Quality Assurance/Quality Control
RPD	Relative Percent Difference
RL	Reporting Limit (Quantitation Limit)
SOGs	Standard Operating Guidelines
SOPs	Standard Operating Procedures
TOC	Total Organic Carbon
Town	Town of Weymouth, Massachusetts
µg	Microgram
µS	Microsiemen
USEPA	United States Environmental Protection Agency
USEPA-NE	USEPA-New England (Region 1)
VOCs	Volatile Organic Compounds
WPA	Massachusetts Wetlands Protection Act

1.0 PURPOSE AND DESCRIPTION

Whitman's Pond is an approximately 210-acre waterbody that is divided into three basins, including the Main Basin, the West Cove, and the South Cove. The South Cove is used to supplement the Town's water supply. The West Cove is former swampland that is connected to the Main Basin by a culvert during high water but is isolated during drought periods.

The growth of exotic invasive weeds such as fanwort (*Cabomba caroliniana*), variable-leaf milfoil (*Myriophyllum heterophyllum*), and curly-leaf pondweed (*Potamogeton crispus*) plagues Whitman's Pond at nuisance levels. In the recent past, herbicide treatments with fluridone (trade name Sonar) and hydroraking have been conducted in an attempt to control excessive macrophyte growth in the West Cove and a portion of the Main Basin. Herbicide treatment of the entire Main Basin was not permitted by the Weymouth Conservation Commission due to concerns about impacts to spawning and juvenile development of anadromous alewife (*Alosa pseudoharengus*).

ESS was contracted by the Town to develop a Vegetation Management Action Plan for Whitman's Pond. The primary purpose of this project is to synthesize existing data and collect new data to fill data gaps in order to develop management recommendations within the Vegetation Management Action Plan. The Whitman's Pond Working Group desires that the Vegetation Management Action Plan be based on sound science with the primary goals of protecting or improving the following:

- the Town water supply
- the ecological value of the pond (with particular attention to alewife habitat)
- recreational opportunities

This QAPP has been developed to ensure that all data is collected in a sound and standardized manner with appropriate QA/QC.

In accordance with USEPA guidelines, Table A summarizes the location of information required in this QAPP.

Table A. Required Information Checklist

USEPA-NE Worksheet No.	Worksheet Title	Location in QAPP
1	Title and approval	Prior to table of contents
2	Table of contents & document format	In narrative
3	Distribution list	Prior to table of contents
4	Project personnel sign-off sheet	Relevant personnel are included on the approval page
5a	Organizational chart	Figure 1
5b	Communication pathway	Section 2.1
6	Personnel responsibilities and qualifications	Section 2.3 and Appendix A
7	Special personnel training requirements	Section 2.4
8a	Project scoping meeting attendance sheet, agenda	Section 3.1
8b	Problem definition/site history & background	Section 3.2
9a	Project description	Section 1.0
9b	Contaminants of concern	Section 6.0
9c	Field & QC sample summary	Section 7.5
10	Project schedule timeline	Section 4.0

Table A. Required Information Checklist

USEPA-NE Worksheet No.	Worksheet Title	Location in QAPP
11a	Project quality objectives/decision statements	Section 7.0
11b	Measurement performance criteria	Section 7.0
12a	Sampling design & rationale	Section 5.0
12b	Sampling locations, methods, SOP requirements	Section 5.0
13	Project sampling SOP references	Appendix B (SOGs)
14	Field sampling equipment calibration	Appendix B (SOGs)
15	Field equipment maintenance, testing and inspection	Appendix B (SOGs)
16	Sampling handling, tracking, custody	Section 6.0
17	Field analytical method/SOP references	Section 6.0
18	Field calibration	Appendix B (SOGs)
19	Field maintenance	Appendix B (SOGs)
20	Fixed lab. Analytical method/SOP references	Appendix C (Laboratory QAP and SOPs)
21	Fixed lab. instrument maintenance & calibration	Appendix C (Laboratory QAP and SOPs)
22a	Field sampling QC	Section 7.5
23	Field analytical QC	Section 7.5
24	Fixed laboratory analytical QC	Appendix C (Laboratory QAP and SOPs)
25	Non-direct measurement criteria	Section 9.0
26	Project documentation and records	Sections 8.0 and 14.0
27a	Assessment and response	Section 10.0
27b	Project assessment	Section 10.0
27c	Project assessment plan	Section 10.0
28	QA management reports	Section 11.0
29a	Data evaluation process	Section 13.0
29b	Data validation summary	Section 13.0
29c	Data validation modifications	Section 13.0
30	Data usability assessment	Section 10.0

2.0 PROJECT MANAGEMENT

ESS has been contracted by the Town to prepare a Vegetation Action Management Plan for Whitman's Pond. Carl Nielsen will be the ESS Project Manager and also serve as the project internal QA Officer. The Project Manager will be responsible for coordinating all field and laboratory efforts as well as serving as a direct contact for all parties involved with the project. Responsibilities of the QA Officer will be primarily associated with ensuring that personnel serving the project are properly trained in all appropriate procedures relating to sample collection and data generation. The QA Officer will regularly verify that the items described in this QAPP are being followed. Additionally, the QA Officer will verify conformance with project reporting deadlines and data quality objectives, and ensure that project deliverables satisfy contract provisions.

This QAPP will direct field and laboratory activities for the Vegetation Action Management Plan at Whitman's Pond. ESS will be responsible for data generation and acquisition as appropriate. Data acquisition will include reviewing existing studies, sample collection and field assessments. Premier Labs, Inc., a Massachusetts certified laboratory, will provide analytical services for all sediment bulk chemical and water quality parameters (except those analyzed in the field by ESS personnel). GeoTesting Express will provide analytical services for bulk physical sediment samples.

The project organizational chart (Figure 1) describes the principal officials and investigators that are associated with the project and illustrates communication pathways.

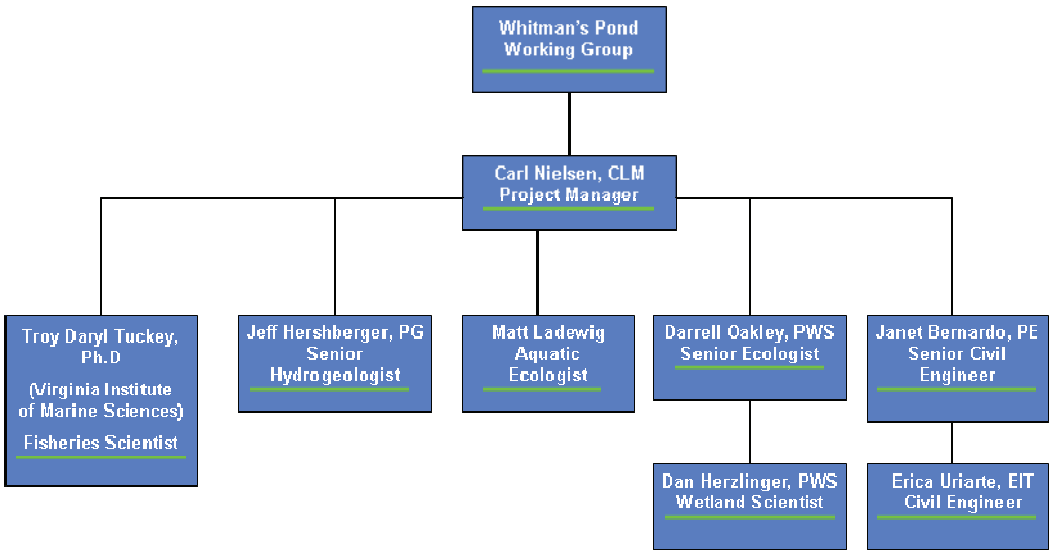


Figure 1. Organizational Chart for Whitman's Pond Vegetation Management Action Plan

2.1 Communication Pathways

For all work requested by the Town, Carl Nielsen of ESS will serve as Project Manager and will coordinate all field and office work to ensure that it meets the standards established for the project and that work is performed in a timely manner. Mr. Nielsen will also act as Quality Assurance Officer and will review fieldwork, lab reports, and client deliverables for acceptability. Mr. Nielsen will ensure that all involved personnel are properly trained in appropriate protocols and will review reports for accuracy and completeness. In addition, Mr. Nielsen will provide regular progress updates to Mary Ellen Schloss, the Project Supervisor from the Town and the Whitman's Pond Working Group, and will be responsible for meeting all project requirements. Mr. Nielsen will serve as the primary point of contact for the entire project.

Field data collection will be conducted by Dan Herzlinger and Matt Ladewig of ESS. They will be responsible for conducting all field work at Whitman's Pond and developing reports. These staff will report directly to Mr. Nielsen.

Senior ESS staff including Janet Bernardo, Jeffrey Hershberger, and Darrell Oakley may assist Mr. Nielsen with reporting oversight and engineering feasibility on the project. They will coordinate with the field data collection team, as needed, and report to Mr. Nielsen.

GIS data management and mapping will be overseen by Matt Ladewig of ESS. He will ensure that all GIS work completed is accurate and appropriately presented.

Dr. Troy Tuckey of the Virginia Institute of Marine Science will advise the project team on river herring ecology to ensure that management actions do not negatively impact the alewife run associated with Whitman's Pond. Dr. Tuckey will review newly acquired data and data available from previous studies and report to Mr. Nielsen.

2.2 Modifications to the QAPP

In the event that the QAPP requires substantial modification, Carl Nielsen will contact the Project Supervisor from the Town before proceeding with any further project activities. The organizational chart (Figure 1) describes the principal officials and investigators associated with the project and illustrates the chain of communication and authorization.

2.3 Personnel Responsibilities and Qualifications

A summary of personnel responsibilities is presented below. Individual resumes for each member of the project team are presented in Appendix A.

- **Carl Nielsen, CLM, Senior Water Resource Scientist/Aquatic Biologist.** Mr. Nielsen is a Certified Lake Manager and has an MS degree in Fisheries and Wildlife. He has over 20 years of experience in aquatic ecosystem assessment and management. Mr. Nielsen has been personally responsible for conducting over 50 lake and pond diagnostic/feasibility assessments, many of which he has used to develop comprehensive lake and watershed management plans. He has also specialized in the investigation and management of water quality related problems and nuisance aquatic vegetation. This will ensure that the ESS Team will be able to provide the Town with the most appropriate plant management solutions at Whitman's Pond. Mr. Nielsen has led more than 150 aquatic resource studies ranging in size from small pond and stream systems to analyses of entire watershed systems. For this project, he will serve as the primary point of contact, attend project meetings, manage and oversee fieldwork, and prepare the final report.

- **Jeffrey Hershberger, PG, Senior Hydrogeologist.** Mr. Hershberger is a professional geologist with over 20 years of experience and an MS degree in Geology. Mr. Hershberger's professional experience emphasizes groundwater/surface water interactions and development of conceptual site models of hydrogeology and subsurface transport. Related field experience includes subsurface investigations, multi-media sampling events, water supply exploration, and aquifer testing programs. For this project, Mr. Hershberger will be responsible for the hydrologic evaluation of Whitman's Pond and assist with the water supply resource assessment.

- **Darrell Oakley, PWS, Senior Ecologist.** Mr. Oakley is a Professional Wetland Scientist with over 16 years of experience in wetlands science (mapping, delineation, and assessment), wildlife habitat and rare species assessments, water quality assessment, watershed management, and regulatory compliance. He has participated in numerous watershed, wetland, and wildlife studies. For the Whitman's Pond project, Mr. Oakley will be responsible for leading wetland and wildlife field surveys and data gathering as well as with evaluating the potential impacts of any proposed management actions.

- **Dan Herzlinger, PWS, Wetland Scientist.** Mr. Herzlinger is a Professional Wetland Scientist with over eight years of experience conducting ecological field studies, wetland delineations, environmental permit review/preparation, natural resource site assessments, environmental inspection/construction oversight, wildlife habitat evaluations and rare species surveys. His range of project experience includes the siting and permitting of energy generation facilities and infrastructure, commercial development, lake management and watershed assessments for non-point source pollution. Mr. Herzlinger has expertise in the use of GIS, sub-meter accuracy GPS, laser rangefinder and methodology for conducting visual assessments. He also has a strong working knowledge of the

WPA and its implementing regulations (310 CMR 10.00). As the Conservation Agent for the Town of Acushnet, Massachusetts, Mr. Herzlinger oversaw the administration and enforcement of the WPA. He will be responsible for assisting Mr. Oakley with wetland and wildlife assessments for the Whitman's Pond project.

- **Janet Carter Bernardo, PE, Senior Civil Engineer.** Ms. Bernardo is a registered professional civil engineer with over 21 years of technical and management experience in civil site design. As a Project Engineer and Manager, Ms. Bernardo has managed and participated in numerous site designs and permitting projects, including aquatic restoration, storm water, and dredging projects. These projects include storm water management, public boat access, zoning analysis, traffic analysis, drainage and utility design, construction details, and specifications. Specializing in storm water remediation, she is also experienced in local and state permitting and has served as the reviewing consultant for various Massachusetts communities. Ms. Bernardo will be responsible for developing cost estimates and water and sediment volume calculations.

- **Erica Uriarte, EIT, Civil Engineer.** Ms. Uriarte's experience includes civil work such as site grading, earthwork analysis, storm water management system design, utility research and layout, dredging, and subsurface disposal system design. She has assisted in the preparation of various environmental regulatory permit applications in Massachusetts, including Chapter 91 Waterways License applications, 401 Water Quality Certifications, and Notices of Intent. Ms. Uriarte's computer skills include AutoDesk Land Desktop, Hydraflow, and HydroCAD. On this project, she will assist Ms. Bernardo with water and sediment volume calculations and developing cost estimates for any management actions requiring engineering.

- **Matt Ladewig, CLM, Aquatic Ecologist.** Mr. Ladewig is an aquatic ecologist with experience in lake management, fish ecology, water quality assessment, rare species surveys, and sediment sampling. He has studied over 40 lakes and ponds to date and has been directly involved in all phases of developing plan management plans, including field work, laboratory work, data analysis, modeling, and reporting. Mr. Ladewig is certified by the Society for Freshwater Science as a macroinvertebrate taxonomist and is approved as a Certified Lake Manager by the North American Lake Management Society. His primary role on this project will be to coordinate and lead necessary field surveys and assist Mr. Nielsen with the review of existing data and development of final management recommendations. Mr. Ladewig will also be responsible for ensuring that high-quality GIS-based maps are produced that will facilitate the restoration effort at the pond as the Town seeks permits and regulatory approvals moving forward.

- **Troy Daryl Tuckey, Ph.D, Fisheries Scientist.** Dr. Tuckey is affiliated with the Virginia Institute of Marine Science where he conducts scientific research related to fisheries population dynamics and applies the results to solve management problems. His primary research interest is diadromous fishes including river herring, American shad, and American eel, with a particular focus on the critical early life stages. Dr. Tuckey has collected specimens of American shad, hickory shad, alewife, and blueback herring ranging from larvae to adults and performed age, growth, and reproductive studies to understand population dynamics of these species. He chaired the Alosine Species Team for the Maryland Sea Grant Ecosystem Based Fisheries Management initiative where he authored publications examining the life history of shad and river herring as well as water quality issues that affect these species. Dr. Tuckey also served on the Atlantic States Marine Fisheries Commission Shad and River Herring Subcommittee and continues to play an advisory role on the management of these species through the Virginia Marine Resources Commission and the Virginia Department of Game and Inland Fisheries. His primary role on the ESS Team for this project will be to advise the project team on river herring ecology to ensure that management actions do not negatively impact this very important alewife run.

2.4 Special Training Requirements/Certification

The Project Team has extensive experience in water quality and sediment sampling; aquatic plant and bathymetry mapping; macroinvertebrate sampling and taxonomy; fisheries surveys; wildlife assessments and pond and watershed management. Carl Nielsen is a CLM and has over twenty years of experience in limnology and lake management. Matt Ladewig is a certified macroinvertebrate taxonomist and CLM, while Darrell Oakley and Dan Herzlinger are PWSs with training in identification and mapping of aquatic plants. Field staff has special training in the use of the GPS that will be required for project implementation.

No additional special training or certification courses are specifically required for project implementation. However, the Project Team is trained in limnological field methods, including bathymetry mapping, sediment sampling, water quality sampling, fisheries surveys and macrophyte identification from previous academic study, routine participation at conferences on the subject of lake management, as well as during informal ESS in-house training associated with a variety of similar projects throughout New England. Additional in-house training will be provided for ESS staff as necessary.

3.0 PLANNING/PROJECT DEFINITION

3.1 Project Planning Meetings

Initial scoping of this project was defined by the Town in its Request for Proposals for this project. A project "kick-off" meeting was held on April 2, 2012 in order to clarify project goals and contract details. A list of attendees and affiliation is presented in Table B.

Table B. Kick-off Meeting Attendance List

Name	Affiliation
Susan Kay	Mayor, Town of Weymouth
Mary Ellen Schloss	Conservation Administrator, Town of Weymouth
Arthur Mathews	Weymouth Town Council and Whitman's Pond Association
Jeff Bina	Department of Public Works Director, Town of Weymouth
Chip Fontaine	Town Engineer, Town of Weymouth
Jim Clarke	Planning and Community Development Director, Town of Weymouth
George Loring	Conservation Commission Chairman and Herring Warden, Town of Weymouth
Scott Dowd	Conservation Commission, Community Preservation Committee Member, Town of Weymouth
Phil Lofgren	Assistant Herring Warden, Town of Weymouth
Trish Pries	Whitman's Pond Association
Tom Daru	Whitman's Pond Association
Carl Nielsen	ESS Group, Inc.
Matt Ladewig	ESS Group, Inc.

3.2 Problem Definition/Site History and Background

Whitman's Pond is an approximately 210-acre waterbody that is divided into three basins, including the Main Basin, the West Cove, and the South Cove. The South Cove is used to supplement the Town's water supply. The West Cove is former swampland that is connected to the Main Basin by a culvert during high water but is isolated during drought periods. The Main Basin has depths up to 27 feet, while the West and South Coves are shallower with maximum depths of approximately four feet. The pond's

watershed is approximately 13 square miles. The relatively rapid flushing rate at Whitman's Pond has important implications for pond management.

Whitman's Pond suffers from cultural eutrophication and excessive aquatic plant growth. The growth of exotic invasive weeds such as fanwort, variable-leaf milfoil, and most recently curly-leaf pondweed plagues Whitman's Pond at nuisance levels. Fanwort, in particular, has been a nuisance for over three decades. The Town is seeking to abate the current poor conditions in the pond and develop a long-term plan for managing excess aquatic plant growth in Whitman's Pond. Accordingly, the Vegetation Management Action Plan will outline a set of short-term "emergency" management recommendations as well as a suite of long-term management recommendations to address the aquatic invasive plant growth in the pond as part of a sustainable plan. Cost estimates to permit and implement the selected management recommendations will also be provided and the Vegetation Management Action Plan will weigh management options for Whitman's Pond against any potential negative impacts on the pond's ability to provide water supply, fisheries and wildlife habitat, or recreational resources.

In order to provide the Town with management recommendations for Whitman's Pond, the Project Team will review existing and readily available information covering many of the critical physical, chemical, and biological aspects of Whitman's Pond and its watershed. This information will be used to supplement data collected under this QAPP and provide a context for sufficiently documenting the pond's present condition, establishing a set of baseline data, and forming the basis for analysis and management recommendations.

Work will be conducted under the guidance of this QAPP, which is compatible with USEPA and MassDEP guidelines and developed specifically for the Whitman's Pond project. All laboratory water quality and sediment analysis will be performed by a Massachusetts certified laboratory.

4.0 PROJECT DESCRIPTION AND SCHEDULE

This project is designed to establish a set of baseline data, covering key physical, chemical and biological aspects of Whitman's Pond and its watershed. These data will be used to develop a Vegetation Management Action Plan. To this end, the following tasks will be completed:

1. **Kick-Off Meeting** – Attend initial kick-off meeting and site visit to the pond with members of the Whitman's Pond Working Group.
2. **Data Review –** Review existing data and reports to evaluate pond's current condition and identify any data gaps.
3. **Develop a Quality Assurance Project Plan** – Prepare and submit a QAPP to MassDEP and USEPA for approval.
4. **Sediment Sampling** – Measure soft sediment distribution and determine the quality of the pond sediments that may affect ecological health for dredging feasibility.
5. **Fisheries and Wildlife** – Assess fisheries, aquatic macroinvertebrates, mammals and bird communities associated with the pond. Collect water quality samples to assess its suitability to support fisheries and other aquatic life.
6. **In-Lake Vegetation** – Conduct an assessment of aquatic macrophytes with an emphasis on documenting invasive species.
7. **Wetlands Assessment** – Assess wetland communities associated with the pond and potential impacts to wetlands from various management recommendations.
8. **Recreational Use Assessment** – Document current and historic public recreational uses of the pond.

9. **Hydrologic/Hydraulic Assessment and Bathymetry** – Determine the pond's water depth contours. Use existing hydrologic data and updated bathymetry map to assess feasibility of draw-down as management option.
10. **Water Supply Assessment –** Review existing records of water withdrawal from South Cove to Great Pond to assess the usage of this resource and implications for management recommendations.
11. **Feasibility Assessment** – ESS will meet with the Whitman's Pond Working Group to assess the feasibility of the initial short and long-term management recommendations for Whitman's Pond.
12. **Draft Report –** ESS will prepare a draft report of the Vegetation Management Action Plan that includes a description of methodology and results in narrative, tabular, and graphical formats, as appropriate.
13. **Final Report** – ESS will meet with the Whitman's Pond Working Group to discuss comments on the draft report and incorporate these into the final Vegetation Management Action Plan report.

In order to successfully achieve the goals and objectives stated above, project tasks will be completed according to the project schedule (Table C). The project began with a kick-off meeting on April 2, 2012 and is anticipated to be completed by January 2013.

Table C. Project Schedule

Task	2012												2013
	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Jan
Task 1 – Kick-off Meeting											
Task 2.1 – Data Review											
Task 2.2 - QAPP											
Task 2.3 – Sediment Sampling											
Task 2.4 – Fisheries and Wildlife											
Task 2.5 – In-lake Vegetation											
Task 2.6 – Wetlands											
Task 2.7 – Recreational Use											
Task 2.8 – Hydrologic/Hydraulic											
Task 2.9 – Water Supply											
Task 3 – Feasibility Assessment											
Task 4 – Draft Report											
Task 5 – Final Report											

5.0 TECHNICAL DESIGN FOR FIELD SAMPLING

5.1 Hydrologic/Hydraulic and Water Supply Assessment – Pond Bathymetry

Whitman's Pond will be surveyed via sonar or calibrated rod at up to 143 points using a modified point-intercept method (Figure 2). This methodology uses the point-intercept method to set up a mapping framework but allows staff to adjust final point positions based on field observations. This permits staff to optimize the use of field time while creating a more representative bathymetry map than might be otherwise expected. Sonar will be used to measure water depth in deep, weed-free portions of the pond and a calibrated sounding rod or line will be used in shallow or weedy portions of the pond. Horizontal positions will be collected with a Trimble GeoXT DGPS and water levels on the day of survey will be vertically referenced to a mapped control structure in the pond.

Bathymetric information will be incorporated into the hydrologic/hydraulic assessment of Whitman's Pond used to evaluate the feasibility of drawdown as a management option. Bathymetric data will be collected and incorporated into a bathymetric map using methods described in the ESS SOGs for the creation of a GIS map (Appendix B).

5.2 Sediment Sampling

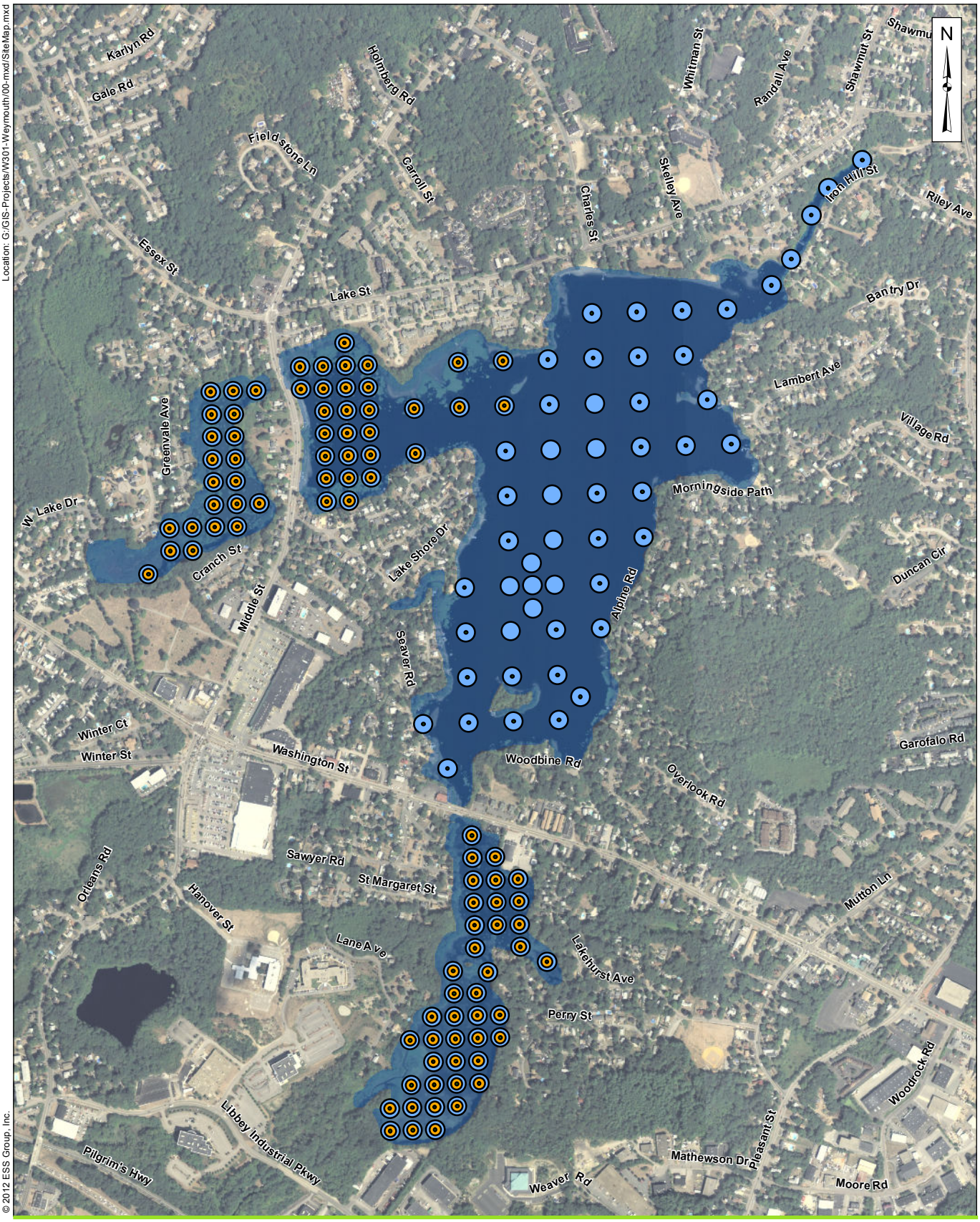
ESS staff will measure soft sediment depth in select areas of the pond to develop a sediment isopach (thickness) map for evaluating dredging feasibility. The targeted areas were selected in consultation with the Whitman's Pond Working Group and include the West Cove, the northwestern cove of the Main Basin, and the South Cove (Figure 2). In these areas, measurements will be made at up to 92 points using a modified point-intercept methodology. This methodology uses the point-intercept method to set up a mapping framework but allows staff to adjust final point positions based on field observations. This permits staff to optimize the use of field time while creating a more representative isopach map than might be otherwise expected. At each point, ESS staff will first measure and record water depth using a calibrated rod (tile probe). Then the tile probe will be driven into the sediment until first refusal is achieved. At this point, the total depth will be recorded, allowing the thickness of the surficial soft sediment layer to be calculated. A description of the underlying sediments (e.g., silt, sand, gravel, hardpan, etc.) will also be recorded. The lateral position of each point will be recorded with sub-meter accuracy using a Trimble GeoXT DGPS. These data will be used to calculate the volume, average depth, and maximum depth of organic matter. A GIS map will be prepared depicting sediment isopach at 1-foot contour intervals throughout the pond.

ESS personnel will collect sediment samples from Whitman's Pond in a manner consistent with the SOGs for Collection of Sediments from Freshwater Environments (Appendix B). Up to 15 sediment cores will be collected in areas of representative soft sediment thickness. MassDEP allows up to three samples to be composited for analysis. Therefore, up to fifteen cores will be analyzed as five composite samples. One composite sample will be collected from each of the three target areas (West Cove, northwestern cove of the Main Basin, and South Cove) with potentially two additional composite samples from the South Cove. The locations of these cores will be located after initial sediment isopach mapping has been completed. Therefore, proposed locations have not been included at this time.

Each of the composite samples will be comprised of three distinct sediment cores that will be homogenized for analysis. The exception will be VOC samples, which will be extracted from a single core prior to homogenization in order to avoid volatilization of the samples. The sediment cores obtained will be logged, photographed, and sampled in the field in order to obtain representative samples for delivery to the appropriate laboratory. ESS will deliver the sediment samples to a state certified laboratory along with required chains. The lab will perform the bulk physical and chemical analysis that would be required for 401 Water Quality Certification as outlined below.

- ***Bulk Physical Analysis:*** Bulk physical analysis of recovered sediments will be performed by GeoTesting Express of Acton, Massachusetts. Composite samples will be analyzed for grain size.
- ***Bulk Chemical Analysis:*** Bulk chemical analysis will be performed by Premier Labs, Inc. on recovered sediment. Three composite samples will be analyzed from the nine cores collected at the pond. Samples will be analyzed for VOCs, heavy metals (arsenic, cadmium, chromium, copper, lead, mercury, nickel, and zinc), total PCBs, EPHs with target PAHs, pesticides, moisture content, and TOC.

Detection limits for this testing will be targeted at a level appropriate for material removal, storage, and disposal as specified under "Regulations for Water Quality Certification for Dredging, Dredged Materials Disposal, and Filling in the Waters of the Commonwealth" and sufficient to complete an application for an Army Corps of Engineers 404 Permit.



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Vegetation Management Action Plan
Whitman's Pond, Weymouth, MA

1 in = 1,000 ft

0 500 1,000 Feet

Source: 1) MassGIS, Orthos, 2009

Legend

- Aquatic Plants
- Isopach
- Bathymetry

Proposed Mapping Locations

Figure 2

Sample material will be preserved in accordance with the specific requirements of the laboratory methods used to analyze each sample.

Records of observations will be kept in a weather-resistant field book. Photographic documentation will be stored electronically.

5.3 Water Quality

ESS will collect a single round of water quality data to establish baseline conditions in the pond. The following parameters will be measured by ESS staff in the field: pH, dissolved oxygen, temperature, specific conductance, turbidity, and color. Data will be collected at the pond surface and bottom (MB-S and MB-B, respectively), at one location in the West Cove (WB) and at the mouth of the Old Swamp River (SB) for a total of four sampling locations (Figure 3). ESS will also measure in-pond water clarity (Secchi depth) and collect temperature and dissolved oxygen data through the water column at the deepest location in the pond. These data will be used to develop a full vertical profile of Whitman's Pond and estimate the areal extent of low oxygen conditions.

In addition to the field parameters, ESS will collect samples for laboratory analysis at each of the four sampling locations. These samples will be analyzed for total phosphorus and total nitrogen by a state-certified laboratory (Premier Laboratory of Dayville, Connecticut). Water quality sampling will be conducted in accordance with ESS SOGs (Appendix B) and laboratory analysis will be consistent with the contract laboratory QAP and SOPs (Appendix C).

As a QA/QC measure for the water quality sampling activities, one duplicate sample will be sent to the laboratory.

Records of observations will be kept in a weather-resistant field book. Photographic documentation will be stored electronically.

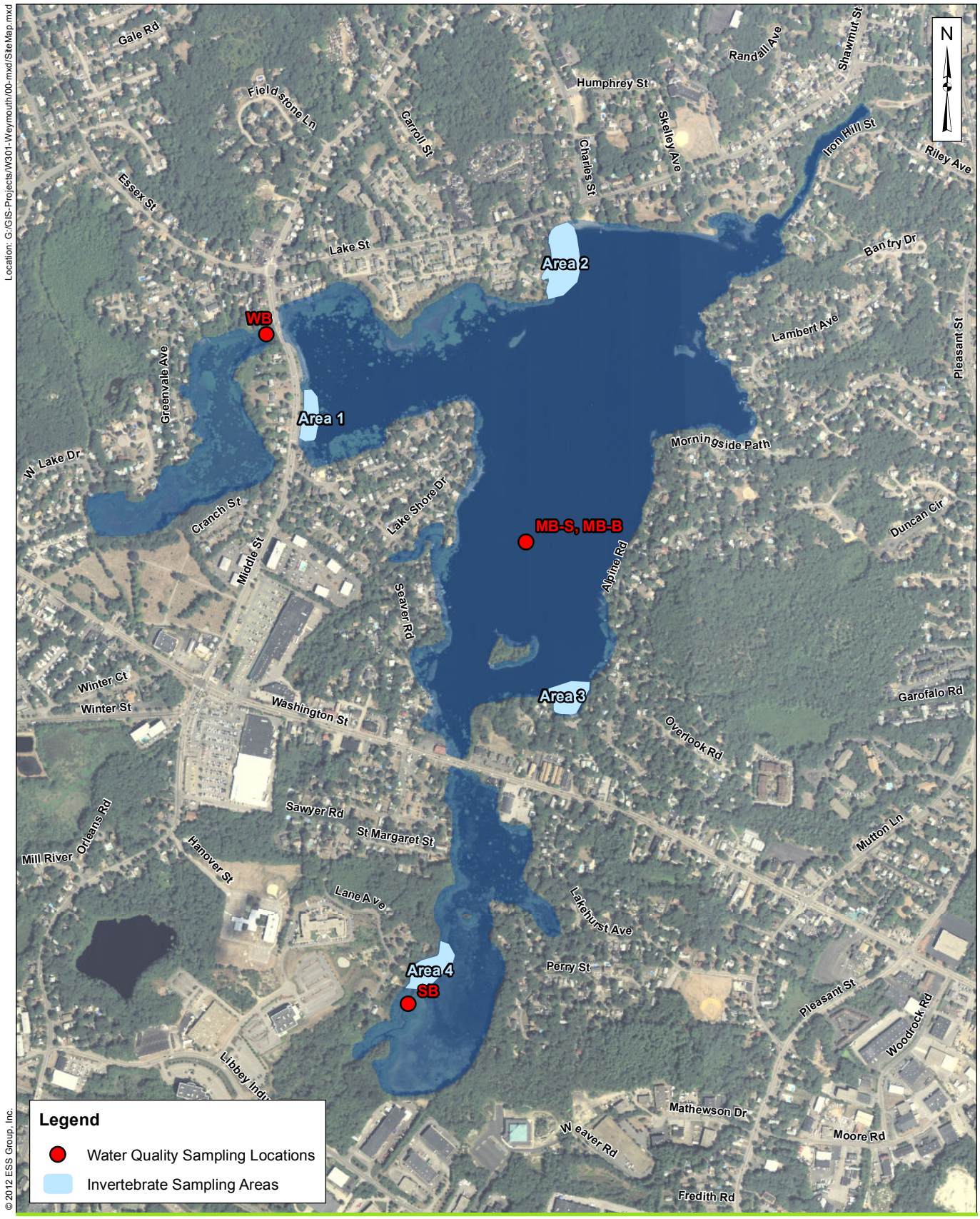
5.4 In-pond Vegetation Assessment

An inventory of the aquatic plant community will be conducted for the purpose of describing species composition, cover, and biovolume during the period of peak development (July to August). These will be assessed from a boat at up to 143 locations using a modified point-intercept method. This methodology uses the point-intercept method to set up a mapping framework but allows staff to adjust final point positions based on field observations. This permits staff to optimize the use of field time while creating a more representative macrophyte map than might be otherwise expected. Mapping equipment may include a Marcum vs625 color underwater camera, a plant rake, and/or a view scope, as needed to characterize growths. Dominant species, percent cover, and biovolume will be recorded at each point and positions will be taken with a Trimble GeoXT DGPS.

Plant species encountered will be identified in the field by a trained and qualified staff member. Specimens that cannot be readily identified in the field will be retained and identified using taxonomic keys, such as *A Guide to Aquatic Plants in Massachusetts* (New England Aquarium 1999), *Aquatic and Wetland Plants of Northeastern North America* (Crow and Hellquist 2000) and a series produced by the New Hampshire Agricultural Experiment Station (Crow and Hellquist 1982).

Maps depicting the distribution of plant cover and plant biovolume will be created in GIS format and the acreage of Whitman's Pond covered by each density or biovolume level of aquatic plants will be determined. The data collected from this study will provide an update to conditions previously documented in the pond and evaluate the potential costs of various plant management techniques for Whitman's Pond.

Vegetation mapping will follow the ESS SOGs for the creation of an aquatic plant map (Appendix B).



Vegetation Management Action Plan
Whitman's Pond, Weymouth, MA

1 in = 1,000 ft

0 500 1,000 Feet

Source: 1) MassGIS, Orthos, 2009

Proposed Sampling Locations

Figure 3

Records of observations will be kept in a weather-resistant field book. Photographic documentation will be stored electronically.

5.5 Fish and Wildlife

The objective of this assessment is not to inventory the species that inhabit Whitman's Pond but rather to characterize the pond's fish and wildlife communities and hydraulically connected wetlands. Although observations will be made during field visits over several months, a full seasonal analysis of habitat use is beyond the scope of this study. As such, direct observation of fish and wildlife will be qualitative and used primarily to supplement previous surveys. Observations will be focused on potential nuisance species (e.g., Canada Goose) as well as those that may be sensitive to particular management options (e.g., drawdown).

Fish Habitat Suitability Assessment

The primary source of habitat suitability information for alewife at Whitman's Pond will be the study currently underway through DMF, which is being conducted under a separate QAPP (Chase 2010). Sediment (substrate), plant cover, and water depth information collected as part of this study will be used to develop a GIS map that depicts probable suitability of pond habitats for species known to inhabit Whitman's Pond. Habitat suitability assessments will be based primarily on the USFWS Habitat Suitability Indices published for individual species but may be supplemented with other readily available habitat suitability information sources, as necessary.

Fish and Wildlife Observations

Qualitative assessments will be made of fish, macroinvertebrates, birds, amphibians, reptiles, and mammals that are observed to use Whitman's Pond or its immediate surroundings.

Direct qualitative observation of fish presence in Whitman's Pond will be made using a Marcum vs625 color underwater camera and minnow traps, beach seines, and/or fyke nets. All fish will be identified in situ.

Qualitative assessment of the aquatic macroinvertebrate community in Whitman's Pond will be completed using a 500-micron D-frame dip net in shallow water (less than six feet) and an Ekman benthic grab sampler and/or Marcum vs625 color underwater camera in deeper water. Sampling will be focused in four key areas of the pond (Figure 3). The primary focus of this survey will be on freshwater mussels, with a secondary focus on any other macroinvertebrates collected. All macroinvertebrate samples will be processed and identified in the field, without preservation and transport back to a lab. Samples will be examined on a white pan and macroinvertebrates using field forceps. A hand lens may be used to assist with macroinvertebrate identification as needed. Macroinvertebrate identification will be conducted by a trained taxonomist to ensure accurate identification to the species/genus level.

Avian and other wildlife use of Whitman's Pond and adjacent habitats will also be qualitatively observed. Mammal tracks, scat, lodges, and dens will be noted and the presence of reptiles and amphibians observed during field work will be noted.

Records of observations will be kept in a weather-resistant field book. Photographic documentation will be stored electronically.

Wetland Characterization

Hydraulically connected wetlands around Whitman's Pond will be identified and characterized by a PWS. The level of characterization will be sufficient to assess the potential for these wetlands to be affected by implementation of pond vegetation management options, including drawdown.

Records of observations will be kept in a weather-resistant field book. Photographic documentation will be stored electronically.

6.0 ANALYTICAL PROCEDURES

Water quality samples and sediment quality samples will be collected in the field by ESS personnel using the appropriate containers and will be preserved as required by the lab. All field sampling will follow an approach consistent with that outlined in the ESS SOGs (Appendix B).

Water quality parameters to be tested by ESS personnel in the field will include the following: pH, specific conductance, turbidity, dissolved oxygen, color, clarity (Secchi depth) and temperature. All field meters will be calibrated in accordance with their respective operator's manual prior to fieldwork and as needed while in the field. In order to avoid cross contamination, field equipment will be rinsed prior to each measurement using distilled water or surface water from the next station. Water quality will be assessed in the field using instrumentation in accordance with the SOGs provided in Appendix B.

Water quality parameters to be analyzed by Premier Labs include total nitrogen and total phosphorus.

Sediment quality parameters to be tested by Premier Labs include the following: arsenic, cadmium, chromium, copper, lead, mercury, nickel, zinc, PCBs, full EPH with target PAHs, VOCs, pesticides, TOC, and percent moisture. Sediment quality parameters to be tested by Geo Testing Express will include grain size.

All samples sent to the laboratory for analysis will be accompanied by a completed and signed chain-of-custody.

The laboratory testing programs for surface water quality and sediment quality are summarized in Table D below.

Table D. Water and Sediment Laboratory Parameters

Parameter	Sample Matrix	Number of Samples	Minimum Volume Needed	Sample Container	Sample Preservation	Maximum Hold Time	EPA # (or Equivalent)
Total Phosphorus	Surface Water	5*	250ml	Plastic	H_2SO_4, Ice	28 days	365.1
Total Nitrogen	Surface Water	5*	500ml	Plastic	H_2SO_4,,Ice	48 hours	351.1/ SM4500-NO3 F
Arsenic	Sediment	3	100g	Glass	Ice	6 months	6010B
Cadmium	Sediment	3	100g	Glass	Ice	6 months	6010B
Chromium	Sediment	3	100g	Glass	Ice	6 months	6010B
Copper	Sediment	3	100g	Glass	Ice	6 months	6010B
Lead	Sediment	3	100g	Glass	Ice	6 months	6010B
Mercury	Sediment	3	100g	Glass	Ice	28 days	7471
Nickel	Sediment	3	100g	Glass	Ice	6 months	6010B
Zinc	Sediment	3	100g	Glass	Ice	6 months	6010B
VOCs	Sediment	3	100g	VOA Vial	Methanol, Ice	28 days	8260B
PCBs	Sediment	3	100g	Amber Glass	Ice	7 days	8082
EPH	Sediment	3	100g	Amber Glass	Ice	14 days	MassDEP EPH Method
Pesticides	Sediment	3	100g	Glass	Ice	14 days	8081A

Table D. Water and Sediment Laboratory Parameters

Parameter	Sample Matrix	Number of Samples	Minimum Volume Needed	Sample Container	Sample Preservation	Maximum Hold Time	EPA # (or Equivalent)
Grain Size	Sediment	3	1,000g	Plastic Bag	None required	Indefinite	ASTM D 422-
Percent moisture	Sediment	3	100g	Glass	Ice	14 days	160.3
TOC	Sediment	3	100g	Glass	Ice	7 days	Lloyd Kahn

*Includes a single field duplicate.

The contract laboratories (Premier Lab and GeoTesting Express) routinely analyze duplicate samples for each analytical batch, as part of their internal QA/QC program. Additionally, a single water quality field duplicate will be collected for this project. ESS will conduct internal checks on the validity of field data and will evaluate data as it is received from the lab. If any data is questionable, ESS will contact the lab immediately to determine whether the problem is due to a transcription error or, if necessary, have the lab re-run the sample test.

Table E summarizes the parameters to be measured in the field with respective EPA methods. Specific conductance, dissolved oxygen, temperature, and pH will be measured directly in the water column, where possible. Turbidity and color will be collected in glass or plastic containers and measured immediately in the field.

Table E. Water Quality Field Parameters

Parameter	Specific Conductance	Dissolved Oxygen	Turbidity	pH	Temperature	Color
Sample Matrix	Water	Water	Water	Water	Water	Water
Number of Samples	4	4	4	4	4	4
Sample Container	Instrument	Instrument	Instrument	Instrument	Instrument	Kit
Hold Time	In Field	In Field	In Field	In Field	In Field	In Field
EPA Number	120.1	360.1	180.1	150.1	170.1	NA (Color disc/APHA Platinum – Cobalt)
Expected Range of Field Measurements	0 to 1,500 µS	0 to 15 mg/L 0 to 150 % Sat.	0 to 1000 NTU	4 - 10 SU	-2 to 30 ^{o}C	0 – 500 units
Precision	1% full scale	0.01 mg/L 0.1 % Sat.	NTU (Expected)	0.1 SU	0.1 ^{o}C	NA
Accuracy	$\pm$ 1 % full scale	$\pm$ 0.3 mg/L $\pm$ 2 % Sat.	$\pm$ 2 %	$\pm$ 0.1 SU	$\pm$ 0.2 ^{o}C	NA

7.0 DATA QUALITY OBJECTIVES AND MEASUREMENT PERFORMANCE CRITERIA

High quality data is the goal of all program laboratory analyses, field analytical, and sample collection procedures. Specific data quality objectives have been set for laboratory and field analytical procedures for precision, accuracy, comparability and completeness. If data do not meet these data quality

objectives, they will either not be used or will be explicitly accompanied by a qualifier that describes the nature and degree of deviation.

7.1 Precision

Precision is a measure of the degree to which two or more measurements of the same sample are in agreement as well as a measurement of random error. Precision will be assessed through the measurement of duplicate samples (one sample split into two replicates) and subsequent calculation of the relative percent difference as follows:

$$RPD = \frac{|\text{Result of Duplicate 1} - \text{Result of Duplicate 2}|}{\text{Average of Duplicate 1 and Duplicate 2}} \times 100$$

Objectives for laboratory precision are located in the analyte-specific SOPs located in Appendix C. Objectives for field measurements are located in Table F.

7.2 Accuracy

Accuracy is an evaluation of the degree to which a measured value and a known reference value or true value are in agreement. This is a measurement of systematic error and is often referred to as "bias". Accuracy is determined by the analysis of reference material and comparison of the resulting value to that of the accepted value. The difference between the accepted and reference value is the percent difference. The percent difference is calculated as follows:

$$\text{Percent Difference} = \frac{|\text{Known Value of Reference Material} - \text{Calculated Value of Reference Material}|}{\text{Known Value of Reference Material}} \times 100$$

Objectives for accuracy are located in the analyte-specific SOPs (Appendix C).

7.3 Comparability

Laboratory analytical procedures for each analyte are based upon known and accepted methods, which are detailed in the analyte-specific SOPs (Appendix C).

7.4 Completeness

Completeness is a measure of the amount of valid data obtained compared to the amount that was expected to be obtained under normal conditions. Greater than 80% completeness of laboratory and field analytical samples is expected. Completeness is calculated as follows:

$$\text{Completeness} = \frac{\text{Number of Valid Measurements}}{\text{Number of Measurements Planned}} \times 100$$

Table F. Data Quality Objectives for Field-measured Parameters

Parameter	Specific Conductance	Dissolved Oxygen	Turbidity	pH	Temperature	Color
Sample Matrix	Water	Water	Water	Water	Water	Water
Precision	± 5%	± 5%	± 10%	± 5%	± 5%	± 20%
Accuracy	± 10%	± 10%	± 20%	± 10%	± 10%	NA

7.5 Quality Control

QC requirements are the system of technical activities that measure the performance of a process and will be utilized for field and laboratory analysis. A summary of quality controls to be utilized in the present study is provided in the following sections.

7.5.1 Hydrologic/Hydraulic and Water Supply Assessment – Bathymetry Mapping

By ensuring that the field bathymetry mapping plan is followed by navigating to pre-determined sampling locations using sub-meter accurate GPS and creating GIS figures using SOGs (Appendix B), ESS will be certain to collect and report bathymetry data that are representative of the actual water depths in Whitman's Pond.

7.5.2 Sediment Sampling

By ensuring that the field sampling plan is followed, proper sampling techniques are used, proper analytical procedures are followed, and that sample holding times are not exceeded, ESS will be certain to collect and report water quality data that are representative of actual sediment conditions. All sediment cores will be logged and photographed at the time of collection.

7.5.3 Water Quality Sampling

By ensuring that the field sampling plan is followed, proper sampling techniques are used, proper analytical procedures are followed, and that sample holding times are not exceeded, ESS will be certain to collect and report water quality data that are representative.

The water quality sampling program has been designed to provide data representative of total nitrogen and total phosphorus in the pond. In addition, water quality parameters including temperature, specific conductance, turbidity, pH, dissolved oxygen, water clarity, and color will be measured in the field.

All equipment used in the field efforts will be calibrated, and data will be recorded in a consistent fashion. Duplicate field measurements of a single sample will be performed at a rate of approximately 5% and should agree within 10%. In general, if a discrepancy of greater than 10% is observed between the sample and its duplicate, the piece of equipment will be recalibrated and the sample will be reassessed.

The accuracy, precision, and sensitivity of laboratory analytical data are critical to achieving the QC acceptance criteria of the analytical protocols. With respect to parameters tested in the laboratory, QC requirements for precision, accuracy, and measurement range will be implemented according to Premier Labs' Quality Assurance Plan and GeoTesting Express' Quality Assurance Plan.

Duplicate water quality samples for lab analysis will be collected at a rate of 5% and should agree within 20%. In general, if a discrepancy of greater than 20% is observed between the sample and its duplicate, ESS will request that the lab reanalyze the sample for the analyte in question.

7.5.4 In-pond Vegetation Assessment

Aquatic plant identification will be conducted by a trained and qualified ESS staff member. Plants that cannot be easily identified within the field due to either condition or development stage will be labeled, retained, and transported back to the lab in plastic bags for identification and/or verification using appropriate taxonomic keys, dissecting microscopes, and consultation with other ESS plant experts, as necessary. This will ensure that identifications made are as accurate as possible. Additionally, specimens may be photodocumented, as necessary.

7.5.5 Fish and Wildlife Assessment

Fish and wildlife observations will be made to the lowest practicable taxonomic level based on the information available. Identification will be made by qualified staff that are familiar with the organisms in question. Field guides may be used to assist with identification of avian, fish, or other wildlife species and taxonomic keys may be used for macroinvertebrate identification. No voucher specimens will be retained.

Fish habitat maps will be created in GIS and will be based on information obtained through quality controlled bathymetry mapping, sediment sampling, and in-pond vegetation assessment tasks.

The identification and characterization of hydraulically connected wetlands will be completed by a registered PWS. Although other staff may assist with data collection, the PWS will review and finalize all observations for quality assurance purposes.

8.0 DATA VALIDATION AND MANAGEMENT

Carl Nielsen, the Project Manager, will be in charge of ensuring the proper collection of data and preparation of tables and figures for the entirety of the project. The data will be compiled in Microsoft Excel and the narrative will be written in Microsoft Word format. Other data files (e.g., photos) will also be made available to the Town.

8.1 Field Data

A permanently bound notebook with waterproof pages will be maintained for field sampling. Corrections will be made using a single line through the mistake with the initials of the individual who made them. Entries will include sampling location, time, date, weather conditions, personnel, parameters to be measured and associated data, as well as any problems encountered during sampling. Copies of data sheets will be checked regularly by the Project QA Officer and will be made available for review upon request.

8.2 Laboratory Data

Analytical results will be recorded in a laboratory notebook, specific for each instrument and method. The automated analytical equipment will have computer generated analytical runs and any problems associated with the analytical runs will be flagged and noted. If any corrective action is taken, it will be noted in narrative in the instrument notebook.

The laboratory will provide ESS with the following deliverables:

- Sample data results for all field samples
- Internal and field duplicate sample results, as applicable
- A case narrative of any deviations from QA/QC criteria and observations about the samples that potentially affect sample or data quality (i.e., missed holding times, broken or leaking bottles, and reference standards or check standards outside criteria, etc.).

The following deliverables will not be required, but will be maintained by the laboratory as applicable and made available upon request:

- All raw data
- Duplicate laboratory recoveries and acceptance limits
- Matrix spike/matrix spike duplicate results and acceptance limits
- Method/reagent blank results

- Calibration standards/reference standards/LFB reports
- Copies of instrument logbooks
- Copies of internal chains of custody

All reports will be generated in digital form and available as hard copy, as needed.

9.0 NON-DIRECT DATA ACQUISITION REQUIREMENTS

This section describes protocols associated with data obtained from external sources (i.e., not collected during sampling). A range of readily available data and reports will be used to create a summary of the historical and current condition at Whitman's Pond. This will include a review of previous pond and watershed reports, fisheries data, information provided by the Whitman's Pond Working Group or Town, and external GIS data layers available through MassGIS. External data will be vetted for reliability and applicability to the development of the Whitman's Pond Vegetation Management Action Plan. External data sources that are not clearly documented or are determined to be of questionable quality or applicability will not be used as the sole source for recommending management actions at Whitman's Pond (although they may be used to support or provide context for more reliable data sources). Direct field-based sampling conducted under this QAPP will be used to fill data gaps (or data quality gaps) in existing information sources and help develop recommendations for successful management actions at the pond.

10.0 ASSESSMENT AND RESPONSE ACTIONS

The QA Officer will provide oversight for each field data collection effort to ensure that protocols described in this QAPP are being followed. This duty includes ensuring that field equipment is properly calibrated, data are recorded in a consistent manner, and samples arrive at laboratories in a timely fashion.

The Project Manager will review the final report to ensure that appropriate methodology is adhered to and reported data is within the accepted range for each parameter. Any "outlier" data discovered will be reported in the final report, and potential sources of error will be described and excluded, if deemed non-compliant.

If less than 80% of the data are judged valid by the Project Manager, best professional judgment will be used to verify whether the remaining data are sufficient to complete the Vegetation Management Action Plan. Any limitations of the data set will be communicated to the Town in the draft and final reports prepared for the project.

11.0 QUALITY MANAGEMENT REPORTS

Quality management reports serve to ensure that ESS and the Town are regularly informed on the project status. To accomplish this goal, ESS will maintain regular contact with the Town, subconsultants and vendors, either through telephone, email, or in-person meetings. Additionally, up to four meetings between ESS and the Town have been incorporated into the project Scope of Work to ensure that the project commences, progresses, and terminates in an acceptable manner.

12.0 VERIFICATION AND VALIDATION REQUIREMENTS

Data review, validation, and verification provide methods for determining the usability and limitations of data, as well as a standardized data quality assessment. ESS will be responsible for reviewing laboratory reports for completeness, correctness, and adherence to QC requirements. The Project Manager from ESS will review data received from the laboratories, to assess the data against applicable acceptance criteria. The laboratories conducting the analyses will conduct internal data verifications before submitting the data to ESS.

13.0 VERIFICATION AND VALIDATION PROCEDURES

All field notebook entries, chain-of-custody forms, and other records will be reviewed by the Project Manager for completeness and correctness. Analytical data provided by the laboratories will be reviewed and validated internally to provide information on whether data are acceptable. The Project Manager will be responsible for reviewing the laboratory reports and data packages, as well as data entries and transmittals, for completeness and adherence to QC requirements.

14.0 REPORTING

A draft report will be prepared and submitted to the Town for review and comment. In the draft report, a brief narrative of methodologies used and analytical results obtained will be provided. Tables and figures will also be provided to summarize the findings of the bathymetry, sediment mapping, water quality, in-pond vegetation, and fish and wildlife assessments. Results will be presented in a comprehensive final report, which will incorporate the comments of the Town. The Final Report will be a Vegetation Management Action Plan with prioritized recommendations of corrective actions and their respective estimated costs for managing the pond in a way that is broadly protective of Whitman's Pond resources, particularly alewife habitat and water supply.

15.0 LITERATURE CITED

Chase, B.C. 2010. Quality Assurance Program Plan (QAPP) for Water Quality Measurements Conducted for Diadromous Fish Habitat Monitoring. Version 1.0, 2008 – 2012. Massachusetts Division of Marine Fisheries Technical Report TR-42.

Crow, G.E. and Hellquist, C.B. 1982. Aquatic Vascular Plants of New England. New Hampshire Agricultural Experiment Station, University of New Hampshire, Durham, New Hampshire.

Crow, G.E. and Hellquist, C.B. 2000. Aquatic and Wetland Plants of Northeastern North America. University of Wisconsin Press, Madison, Wisconsin.

New England Aquarium, 1999. A Guide to Aquatic Plants In Massachusetts. New England Aquarium, Central Wharf, Boston, Massachusetts.

Appendix A

Project Team Resumes

CARL D. NIELSEN, CLM
Vice President and Senior Water Resources Scientist

Experience
ESS Group, Inc.:1998 to present

Years of Prior Related Experience: 8

Education
MS, Fisheries and Wildlife, University of Missouri - Columbia, 1994

BA, Biology, Colgate University, 1990

Tufts University, Water Quality Modeling for TMDLs, 40-hr. Workshop, 2001

Professional Registrations and Affiliations
North American Lake Management Society – Certified Lake Manager (CLM)

New England Chapter – North American Lake Management Society

Society for Freshwater Science

Northeast Aquatic Plant Management Society

Qualifications

Mr. Nielsen has over 21 years of experience in the assessment and evaluation of marine and freshwater ecosystems. Mr. Nielsen uses his knowledge of water chemistry and biology to go beyond basic assessments that just identify whether a waterbody is meeting the regulatory standards. Mr. Nielsen has worked extensively in identifying and understanding the ecology of most aquatic organisms including aquatic plants, algae, zooplankton, aquatic invertebrates, fish, reptiles and amphibians. By understanding the ecological needs of the organisms present in an aquatic system Mr. Nielsen is able to tailor management recommendations and mitigation strategies that are appropriate and viewed favorably by the community and most permitting authorities. Mr. Nielsen is also actively involved in the restoration of aquatic systems and has worked to improve water quality and aquatic habitat conditions in numerous lake and river systems throughout New England. As part of these efforts, Mr. Nielsen regularly uses water quality data collected to develop customized scientific watershed models to assist in locating sources of pollution and to evaluate the potential effectiveness of a variety of watershed management strategies. Mr. Nielsen has been Senior Project Scientist for more than 150 aquatic resource studies which have been performed for numerous clients including: federal, state and local governments, municipal water districts, local lake and watershed associations, industrial facilities, property developers, major corporations, utilities, golf courses, ski areas, and airports.

Representative Project Experience

Quaboag and Quacumquasit Lake Association – Quaboag and Quacumquasit Long Term Management Plan Development and Implementation, Brookfield, East Brookfield, and Sturbridge, MA. Developed with the client a comprehensive Long Term Management Plan for the Quaboag and Quacumquasit lake system, which included efforts to improve water quality, reduce algal growth, and manage rooted weed growth. As part of these efforts, an extensive public education component was developed and advanced by Mr. Nielsen through the QQLA organization and with the three town Conservation Commissions.

Town of West Brookfield – Tributary and Groundwater Assessment for Wickaboag Pond, West Brookfield, MA. Developed and oversaw a tributary and groundwater assessment program for Lake Wickaboag. Work was performed in accordance with a Quality Assurance Project Plan (QAPP) developed for the project and included water quality assessment and hydrologic and nutrient modeling. Recommendations made were included in a comprehensive study report.

Quaboag and Quacumquasit Lake Association – Summary Report and Grant Application Assistance, Sturbridge, MA. Responsible for the synthesis of several decades worth of reports and data with the goal of gaining a better understanding of the chemical and biological changes that have occurred in two lakes during the previous 25 years. The primary goal of the study was to better understand how past management actions have altered the quality of each lake, and most importantly to provide a foundation for future management decisions and for securing potential funding for management actions.

Town of West Brookfield – Sediment Sampling and Pre-Dredging Feasibility Assessment for Wickaboag Pond, West Brookfield, MA. Developed and oversaw a sediment assessment program

designed to evaluate the feasibility of dredging the northern basin of Lake Wickaboag. Work was performed in accordance with standard MassDEP protocols to obtain 20 foot cores of sediment from the lake for laboratory analysis and interpretation by ESS. Based on Mr. Nielsen's assessment, the town has moved forward with their plans for dredging the upper basin of the lake.

Wilcox & Barton, Inc. – Water Quality and Biomonitoring Surveys and Ongoing Monitoring Reporting to Inland Wetlands Commission in Support of Major Retail Development, Guilford, CT. Responsible for designing and implementing a comprehensive biomonitoring program in Spinning Mill Brook adjacent to the construction site for a 155,000 square foot retail development. Work included sampling the fish community, benthic invertebrate community, aquatic habitat, and water quality. Work has been performed for two-baseline years of assessment and is likely to continue annually throughout the construction and operation of the proposed development.

Town of Hopedale – Dredging Feasibility Assessment, Hopedale Pond, Hopedale, MA. Project manager and principal scientist for an extensive pre-dredging evaluation of Hopedale Pond, a 35 acre mill pond in Hopedale, MA that is suffering the effects of eutrophication and in-filling from its watershed. A goal of the study is to evaluate the quantity and quality of sediment in the pond as well as to assess the nutrient, bacteria, and other water quality issues related to ongoing inputs from its watershed. The results of the study will be used to provide the town with management recommendations for restoring this pond to its former condition through dredging. Management recommendations will include a detailed description of existing sources of pollution from its watershed and conceptual engineering designs for solving these issues on a site-by-site basis. The Best Management practices (BMPs) that ESS will be recommending will be designed to be economical yet effective. A focus of the ESS strategy will be to implement or retro-fit Low Impact Design (LID) techniques into the existing watershed landscape.

Massachusetts Department of Conservation and Recreation – Dredging Design and Permitting, Farm Pond, Carlisle, MA. Project manager and principal scientist responsible for the restoration of Farm Pond in Great Neck State Park. Mr. Nielsen has designed the initial baseline assessments, sediment sampling program, and is overseeing the engineering design for the pond's restoration which includes dredging. Mr. Nielsen is also overseeing all permitting on this project. Sediment from the pond will be re-used on the state park property as a landscape amendment.

Massachusetts Department of Conservation and Recreation – Dredging Design and Permitting, Robinson Pond, Agawam, MA. Project manager and principal scientist responsible for the restoration of Robinson Pond in Robinson State Park. Mr. Nielsen has designed the initial baseline assessments, sediment sampling program, and is overseeing the engineering design for the pond's restoration which includes dredging. Mr. Nielsen is also overseeing all permitting on this project. Mr. Nielsen will also be making recommendations to improve the annual management of the pond's winter drawdown program.

Glendale Power Station – Housatonic River Freshwater Mussel Survey, Stockbridge, MA. Designed and implemented a comprehensive survey for rare mussels for the Glendale Power Station in Stockbridge, MA in support of a Federal Energy Regulatory Commission (FERC) re-licensing of their hydro-power facility on the Housatonic River. Field survey was performed in the bypass channel of the hydro-power station on the Housatonic River. In addition, Mr. Nielsen was responsible for filing a Rare Animal Observation Form with the Massachusetts Natural Heritage and Endangered Species Program when evidence of a state-listed mussel species was found in the channel. Summarized the findings of the survey in a report supporting the FERC application.

U.S. Army Corps of Engineers – Mill Pond Pre-Dredging Assessment, Littleton, MA. Designed and implemented an assessment of the biological resources of Mill Pond in order to support the USACE with the dredging of Mill Pond. Work included the assessment of fish and macroinvertebrates in Mill Pond and its tributaries (Reedy Meadow Brook and Beaver Brook) which are all located within the Merrimack River watershed. Fish sampling was performed using boat and back-pack electro-shocking equipment.

Walpole Country Club, Dredging Feasibility Assessment – Allen Pond, Walpole, MA. Designed and oversaw a comprehensive investigation of issues pertaining to sediment transport and deposition at Allen Pond on the Walpole Country Club property in Walpole, MA. Work included storm water sampling, in-pond sediment coring for physical and chemical analysis, age dating of sediment cores, water quality assessment, and recommendations for long-term management of the pond. Following the initial work it was determined that a gravel operation upstream of the course was responsible for a large portion of the sediment that had been deposited within the pond. Mr. Nielsen is now overseeing the dredging design, permitting, and construction efforts to restore the ecological and aesthetic value to the pond.

Massachusetts Executive Office of Environmental Affairs – Water Budget Study. Project manager for preparing water budget reports for 74 watersheds and over 300 individual towns in Massachusetts. The Water Budgets Study includes completing water budget assessments for all basins and communities in Massachusetts and evaluating the potential impacts on streamflow. Responsible for the development of basin and community reports that document the water budget results, present associated summary tables and figures/maps. The reports will be developed using a number of document templates that are programmed to interface with the water budgets database using macros to enable these electronic reports to be "living documents" that are readily updatable as new data become available.

Winchester Country Club – Lake Sediment Assessment for a Water Withdrawal Permit, Winchester, MA. Conducted an evaluation of sediment quality in the Upper Mystic Lake adjacent to the Winchester Country Club with regard to its potential impact to the quality of groundwater withdrawn from a proposed irrigation well located adjacent to Upper Mystic Lake. Designed and oversaw the implementation of the sediment and porewater sampling program at Upper Mystic Lake. Oversaw a risk evaluation of the potential for groundwater withdrawn through sediments of Upper Mystic Lake to mobilize metals contained in the sediments. The predicted groundwater concentrations (and the predicted groundwater concentrations within the source area) were also compared to the MassDEP GW-1 (drinking water) standards and GW-3 standards. All of the predicted groundwater concentrations were found to be less than both the MassDEP standards. The predictions were confirmed by the results of the groundwater sampling from the existing test well which showed that groundwater concentrations continue to be compliant with the MassDEP GW-3 standards. Based on the results of the sediment, surface water, and groundwater sampling program; the analytical modeling performed to predict interstitial pore water concentrations within the lakebed sediments; and groundwater concentrations at the proposed irrigation well indicated that is unlikely that the impaired sediment quality identified within Upper Mystic Lake will have a significant adverse impact on the water quality within the proposed irrigation well.

Town of Norton – Diagnostic and Feasibility Assessment for Management of Lake Winnecunnet, Norton, MA. Responsible for conducting an assessment of Lake Winnecunnet and its watershed which are located within a Massachusetts ACEC (Area of Critical Environmental Concern). The deep-water habitat associated with the lake is threatened by the invasive and exotic plant *Cabomba caroliniana* (fanwort) which has spread throughout the lake to the detriment of native plants and potentially native fauna. The need to manage this situation while protecting the potentially rare or threatened species that exist within the lake required extensive survey of the lake shoreline, the major tributaries to the lake (Canoe River and Mulberry Meadow Brook), and the lake outlet (Snake River). Mr. Nielsen conducted a survey of freshwater mussels, aquatic macroinvertebrates, minnows and young-of-the-year fish, aquatic and semi-aquatic plants, reptiles, and amphibians. Based on these detailed surveys, Mr. Nielsen developed a comprehensive lake and watershed management plan for the Town.

Town of Rindge – Hydrologic and Nutrient Budget Analysis for Lake Monomonac, Rindge, NH. Responsible for using existing data to model the potential impacts to Lake Monomonac from a proposed residential subdivision within its watershed. To do this, Mr. Nielsen first had to establish the hydrologic and nutrient budget for the lake and then determine how this would change due the to the proposed development's features. Based on this analysis, the development was found to be a minimal impact to the

lake. Best Management Practices (BMPs) were proposed that could be incorporated into the proposed project's design to further minimize the potential for impact.

Town of Westford – Baseline Characterization, Drawdown Feasibility Assessment, and Long-term Monitoring Program for Nabnasset Lake, Westford, MA. Project Manager and lead scientist in an investigation of the baseline characteristics of Nabnasset Lake and a hydrologically-linked wetland system known as Shipley Swamp. The purpose of the investigations was to determine the nature of impacts that could be anticipated as a result of a proposed winter lake drawdown for the purpose of controlling nuisance aquatic plants. As part of the baseline assessments, Mr. Nielsen established numerous plant monitoring plots within the wetland, biological monitoring stations within the wetland and lake, and established aquatic plant transects within the lake. These stations are currently being monitored annually to determine the response to drawdown (if any) to allow for immediate management actions to be taken as necessary to prevent significant damage from occurring to the ecosystem. Prepared and filed a Notice of Intent for the control of nuisance aquatic plants at Nabnasset Lake by lake drawdown.

Massachusetts Department of Conservation and Recreation – Assessment and Permitting for In-lake Weed Control at Lake Cochituate, Framingham, Wayland and Natick, MA. Prepared Notices of Intent for submittal to the Towns of Framingham, Wayland, and Natick, Massachusetts for the control of nuisance aquatic vegetation at Lake Cochituate. Proposed measures included the use of herbicides, hand-pulling, diver suctioning, milfoil weevils, water circulation, and benthic barriers to control milfoil and curly-leaf pondweed in the lake.

Massachusetts Department of Conservation and Recreation – Assessment and Permitting of Management Activities (Hydro-raking) at Ruggles Pond, Wendell, MA. Prepared a Notice of Intent for the removal of white water lily (*Nymphaea odorata*) and other nuisance aquatic plants by hydro-raking at Ruggles Pond. Conducted aquatic plant mapping and wildlife habitat evaluations at the pond to quantify the growth of nuisance aquatic plants and assess potential impacts from proposed hydro-raking activities on the aquatic community.

Town of Hinsdale – Diagnostic/Feasibility Assessment of Ashmere Lake and Plunkett Reservoir, Hinsdale, MA. The Hinsdale lakes are located in a Massachusetts area of critical environmental concern. Designed and carried out an assessment of the physical, chemical and biological characteristics of these lakes which included water quality assessment, fish and wildlife evaluations, rare/threatened/endangered species investigations, and wetland plant assessments. The work served as the basis for making recommendations for controlling nuisance aquatic vegetation within the lakes while minimizing the potential to cause adverse effects on sensitive or rare species common to the ACEC and their watersheds.

Neponset River Watershed Association – Neponset River Flow Stressed Stream Habitat Assessment & Fish Passage Evaluations, Boston, MA. Evaluated streamflow augmentation and instream habitat restoration alternatives and recommended enhancements that would restore habitat for macroinvertebrates and a target list of freshwater fish species in six sub-watersheds draining to the East Branch of the Neponset River, a tributary to Boston Harbor. Mr. Nielsen served as the macroinvertebrate expert on a team designated as the "trio of experts" (a fisheries biologist, macroinvertebrate specialist, and stream hydrologist) charged with assessing 12 selected stream reaches within the study area during a variety of flow regimes. Mr. Nielsen was responsible for preparing the final report.

Town of Deering – Hydrologic and Nutrient Loading Analysis for Deering Reservoir, Deering, NH. Evaluated the potential impact to Deering Lake from two proposed residential sub-divisions to be constructed within the Deering Lake watershed. Town officials and local residents expressed concern over the potential for these developments, as well as future developments, to result in excessive nutrient loading to the lake and contribute to a subsequent decrease in water quality. Deering Lake is classified by New Hampshire Department of Environmental Services (NHDES) as an oligotrophic (low productivity)

waterbody. Mr. Nielsen's hydrologic and nutrient loading analysis aided the Town in protecting the quality of the lake and will serve as the basis for evaluating whether the proposed developments, as well as future developments, are compatible with maintaining current in-lake conditions. The modeling effort and report were reviewed and approved by NHDES.

Town of Charlton – Little Nugget Lake Diagnostic/Feasibility Assessment, Charlton, **MA.** Designed and coordinated a diagnostic and feasibility study of Little Nugget Lake and its watershed in order to determine why aquatic vegetation had recently expanded in the pond to nuisance levels and to recommend appropriate management actions. Management for the pond included a limited herbicide treatment of selected weed beds and education of watershed residents through the design and distribution of an educational brochure.

Aquarion Water Company – Biological Survey in Response to Fish Kill, Easton, CT. ESS responded quickly to design and conduct a biological (fish and macroinvertebrates) assessment of numerous sites upstream and downstream of a reported chlorine spill downstream of a water supply reservoir managed by Aquarion Water Company. Work was initiated immediately following reports of a fish kill in order to characterize the true nature of impacts to Mill River and to develop an appropriate remedial response. Although work on this project is ongoing, initial results seem to indicate that the effects of the spill on the macroinvertebrate community was minimal and that a natural recovery of the stream would be expected within a very short period of time. ESS recommended that baseline macroinvertebrate data be collected for other key streams within the watershed so that any future problems within the water supplier's watershed could be easily evaluated.

Burncoat Pond Watershed District – Burncoat Pond, Towns of Leicester and Spencer, MA. Designed and coordinated a diagnostic and feasibility study of the Burncoat Pond and its watershed in order to determine why aquatic vegetation had recently expanded in the pond to nuisance levels and to recommend appropriate management actions. Recommend management for the pond included the implementation of a controlled winter drawdown of the pond and education of watershed residents.

RIDEM, EPA and Tetra Tech, Inc. – Mashapaug Pond TMDL, Providence, RI. Mashapaug Pond has been identified as impaired by excess nutrients and low levels of dissolved oxygen. The Mashapaug Pond watershed is densely developed with a mix of residential, commercial and industrial land uses. The EPA recently agreed to provide federal funding to support the development of a nutrient TMDL for Mashapaug Pond that is to serve as a pilot project for the rest of the region. Responsible for overseeing the design of the study which included the preparation of a QAPP and the implementation of the water quality, aquatic plant, groundwater quality and quantity, and fish tissue sampling programs. The goal of this project was to collect water quality data sufficient for developing a TMDL for the pond. The nutrient TMDL was prepared for RIDEM and subsequently approved by US EPA Region 1. This work also supported the preparation of a bacterial TMDL for the waterbody.

Lake Wickaboag Preservation Association – Lake Wickaboag, West Brookfield, MA. Designed and implemented an evaluation of the quantity and quality of accumulated sediments within this large recreational waterbody. The lake has a long history of algal problems, which have been regularly controlled through copper sulfate treatment rather than by assessing the source of the nutrients that are causing the algal blooms. Concern was also raised that the copper may be accumulating to toxic levels in the sediments of the lake. Consequently, sediment quality was evaluated to determine its potential to influence in-lake water quality and to assess its potential to adversely affect the aquatic biota.

Town of Stoughton – Diagnostic/Feasibility Study for Ames Long Pond, Stoughton, MA. Responsible for designing and conducting a comprehensive diagnostic/feasibility assessment of Ames Long Pond and its watershed. The evaluation included an assessment of in-pond water and sediment quality, storm water runoff, groundwater quantity and quality, and a vegetation survey of pond.

Management recommendations focused on reducing the growth of nuisance aquatic plants and decreasing the nutrient loading to the pond through in-pond and watershed level management actions.

Town of Wrentham – Multi-Lake Diagnostic/Feasibility Assessment, Wrentham, MA. Responsible for designing and conducting an assessment of the physical, chemical and biological characteristics of Lake Pearl, Lake Archer and Mirror Lake in Wrentham, Massachusetts in order to determine the cause of lake eutrophication. A key concern was the potential for the groundwater entering these lakes to be contaminated by septic systems within their watersheds. The investigation focused on answering this question through the use of seepage meters (to measure groundwater quantity) and littoral interstitial porewater sampling (to measure groundwater quality). Results from this study were used to evaluate the potential benefits of installing sewer lines through portions of the watershed.

Town of Wayland – Biological Assessment of Heard and Mill Ponds, Wayland MA. Designed and implemented a diagnostic/feasibility assessment of Heard and Mill Ponds in Wayland, Massachusetts for the purpose of determining effective treatment methods for the control of nuisance aquatic weed growth, and in particular the exotic species water chestnut (*Trappa natans*). Methods of treatment will rely on mechanical harvesting within key areas of the ponds to ensure that the natural plant community will not be disrupted and can continue to provide valuable habitat to fish and wildlife.

Lake Monomonac Association – Drawdown Feasibility Assessment, Winchendon Springs, MA and Rindge, NH. Conducted a feasibility assessment of Lake Monomonac to ascertain the potential effectiveness of lake drawdown as a method for controlling the nuisance aquatic weed variable leaf milfoil (*Myriophyllum heterophyllum*). Based on the potential impacts of drawdown on the surrounding wetlands and the relatively small area of actual plant infestation, drawdown was not recommended as an appropriate control method at the time.

Town of Ayer – Diagnostic Feasibility Assessment of Spectacle Pond, Ayer and Littleton, MA. Designed and Implemented a diagnostic/feasibility assessment of Spectacle Pond in Littleton, Massachusetts for the purpose of determining effective treatment methods for the control of nuisance aquatic weed growth, and in particular the exotic species fanwort (*Cabomba caroliniana*). Two possible methods of treatment were recommended. One feasible method was chemical treatment of the pond with flouridone at a dose specific to the control of fanwort. This precision approach will ensure that the natural plant community will not be disrupted and will continue to provide valuable habitat to fish and wildlife. As second, non-chemical alternative that was recommended was to control nuisance plant beds through the use of hydro-raking equipment in selected areas.

Town of Dartmouth – Nuisance Aquatic Vegetation Management Plan, Lake Noquochoke, Dartmouth, MA. Conducted an assessment of the physical, chemical, and biological conditions within each of the five basins of Lake Noqochoke and the associated watershed for the purposes of recommending measures for controlling excessive aquatic plant growth. Recommendations for plant control were tailored specifically to meet the needs and goals for each of the lake's five basins. Recommendations included herbicides, hydro-raking, and dredging, as well as measures for improving factors within the watershed which have been affecting conditions within the lake.

Quacumquasit Lake Association – Eurasian Milfoil Transport Study, Brookfield MA. Designed an on-going study to document the quantity of Eurasian Milfoil being transported into Quacumquasit Lake from an adjacent waterbody during flow reversals within an inter-basin connector resulting from large rain events. This innovative management technique is designed to minimize the spread of Eurasian Milfoil, a highly invasive exotic weed. Directly responsible for training Lake Association volunteers to implement the required field work associated with this project. Data collected will be used to build a case for closing the existing flow barrier between the two lakes during times of summer flow reversals.

Town of Wellesley – Multi-Year Limnological Monitoring of Morses Pond, Wellesley, MA. Project manager for the multi-year monitoring of in-lake conditions at Morses Pond, a 103-acre lake within a highly urbanized setting. Investigations to date have revealed infestation of the pond by Water Chestnut (*Trapa natans*), an exotic plant that can grow to nuisance levels. Additionally, algal blooms within the pond have become a concern. On-going monitoring and management recommendations are required to ensure proper protection of the Town's public beach.

White Lily Pond Association – Aquatic Plant Community and Water Quality Assessment of White Lily Pond, Otis, MA. Performed a multi-year evaluation of in-lake conditions at White Lily Pond for the purpose of providing management recommendations. The project was undertaken in order to assess the severity of the pond's aquatic vegetation problem and to provide baseline physical, chemical, and biological conditions of the pond.

Town of Littleton – Fish Sampling and Tissue Analysis for PCBs, Littleton, MA. Responsible for performing an assessment to determine the potential risk to human health posed by PCBs reported from Mill Pond. The fish population was sampled from the pond and tissue samples from both large game fish (bass and pickerel) and large bottom feeding fish (sucker and bullhead) were analyzed for PCBs. Although one fish had accumulated PCBs in its tissue, the levels detected were well below the human health benchmark. Dredging of the pond sediments to remove the contaminated sediments and reduce aquatic plant growth is part of the long-term pond restoration program.

Lake Boon Commission – Nutrient Study of Lake Boon, Hudson/Stow, MA. Performed a study of the physical, chemical, and biological features of Lake Boon and its watershed for the Lake Boon Conservation Commission. The study was conducted in response to perceived increases in eutrophication and aquatic plant abundance within the lake. It was determined that high levels of nutrients were entering the lake from storm water and groundwater sources. Recommendations focused on improving storm water quality through implementation of BMPs and improving groundwater quality through increased septic system maintenance. Recommendations for nuisance plant control focused on a combination of chemical control and lake drawdown. Subsequently designed and implemented an evaluation of the potential ecological impacts that may occur as a result of the proposed drawdown. Impacts to reptiles, amphibians, aquatic invertebrates and non-target plant species were assessed.

Town of Groton – Pre-dredging Survey of Thompson Mill Pond, Groton, MA. Conducted a study of Thompson Mill Pond's sediment quantity and quality in preparation for a dredging program. It was determined that the sediment was of a quality that could be properly disposed without undue risk of contaminating downstream resources or areas adjacent to the disposal area.

Publications

Fuller, R.L., B.P. Kennedy and C. Nielsen. 2004. Macroinvertebrate responses to algal and bacterial manipulations in streams. Hydrobiologia 523:113-126.

Nielsen, C. D. and D. L. Galat. 1996. Substrate association by macroinvertebrates in a large, cold-water springbranch. University of Missouri- Columbia.

Schubert, A. L. S., C. D. Nielsen, and D.B. Noltie. 1993. Habitat use and gas bubble disease in southern cavefish (*Typhlichthys subterraneus*). International Journal of Speleology 22(1-4): 131-143

Presentations

Use of Limno-Barriers as a Potential Non-Chemical Alternative for Management of Weed Beds. New England Chapter of the North American Lake Management Society. June 2011.

Marine Benthic Monitoring Needs for Offshore Energy Projects in the United States. The North American Benthological Society. May 2011.

The Benefits of Bio-monitoring for Watershed Assessment. Charles River Watershed Association's Annual Brown-bag Presentation Series. June 19th, 2007.

Lake Management and the 319 Grant: How to Make Your Grant Application Rise Above the Rest. January 27th, 2007. Annual Meeting of the Massachusetts Coalition of Lakes and Ponds.

DNA Ribotyping as a Tool for Bacterial Source Tracking: A Narraganset Bay Watershed Case Study. April 2003. New England Association of Environmental Biologists Annual Conference.

JEFFREY G. HERSHBERGER, P.G.
Senior Scientist

Experience
ESS Group, Inc.: 1998 to present

Years of Prior Related Experience: 9

Education
MS, Geology, University of Massachusetts, 1992

BS, Geology, Juniata College, 1985

Professional Registrations and Affiliations
Professional Geologist Registration, Pennsylvania (PG-002185-G; inactive)

Professional Geologist Registration, New Hampshire (No. 276)

National Groundwater Association - Association of Groundwater Scientists and Engineers

American Water Works Association and New England Water Works Association

OSHA Hazardous Materials for Hazardous Waste Site Workers (40-hour training in accordance with 29 CFR 1910.120[e]), 1989, and annual refresher training

Rhode Island Water Resources Board, Water Allocation Program Advisory Committee, Out-of-Basin Transfer Committee Member, 2003-2004.

Town of Upton, Water and Wastewater Advisory Committee, member, 2003-2008

Town of Upton, Enterprise Fund Committee, member, 2008-2009

Qualifications

Mr. Hershberger's professional experience includes over 20 years of environmental consulting focusing on the assessment of impacts to soil and groundwater resources, hydrogeologic investigations and water supply feasibility evaluations, permitting and development. His experience emphasizes evaluation and quantification of hydrogeologic processes as related to groundwater flow and contaminant transport, aquifer remediation, aquifer yield, capture zone modeling for remedial design and wellhead protection, analysis of the fate and transport of contaminants in the subsurface, and development of conceptual site models of hydrogeology and contaminant distribution. Mr. Hershberger has significant experience at CERCLA National Priority List (NPL) sites as the technical lead or project manager for the RI/FS, Pre-Design and Remedial Action Implementation work phases at sites throughout New England. Project management experience also includes site investigations and feasibility evaluations under various state regulations, complex field investigation and sampling programs and water supply development and groundwater resource assessments.

Representative Project Experience

Town of Westford, Drawdown Feasibility Assessment. Nabnasset Lake, Westford, MA. Senior Hydrogeologist for the drawdown feasibility assessment for the Town of Westford. The purpose of the evaluation was to determine the nature of impacts that could be anticipated as a result of a proposed winter lake drawdown for the purpose of controlling nuisance aquatic plants. The evaluation focused on potential adverse impacts to certain Town of Westford municipal water supply wells located proximal to the lake as well as other potential supply wells located on the lake.

Town of Wrentham, Wrentham Lakes Study. Wrentham, MA. Task Manager for the hydrogeologic assessment and evaluation of potential impacts to existing and proposed municipal water supply wells due to potential sewer installation. The scope of work included: evaluation of available hydrogeologic information from the Town of Wrentham, the US Geological Survey, and the Charles River Watershed Association; collection of groundwater samples from existing monitoring wells and two existing municipal supply wells to evaluate nutrient loading within the unconsolidated aquifer; evaluation of watershed characteristics in conjunction with the concurrent surface water assessment; preparation of a final technical report; and public presentation of findings.

Town of Sharon, Hydrogeologic Evaluation of Potential Municipal Water Supply Sites. Sharon, MA. Project Manager and Senior Hydrogeologist for the ongoing evaluation of potential water supply sites within the Town of Sharon, Massachusetts. The initial phase of the project consisted of the desktop evaluation of five potential well sites using the Massachusetts Department of Environmental Protection (MassDEP) Site Screening Criteria. Based on the results of the initial phase and discussions with the Superintendent of Public Works and various Town boards, four locations were proposed for further evaluation. The second phase, including test drilling and aquifer testing to develop comparable hydraulic data for

each site, is currently underway. As the project has progressed, field investigations have been performed to assess additional potential water supply sites. To support local decisions, Mr. Hershberger also developed and presented a matrix providing a summary of the information collected for each potential site and ranking the sites relative to each other to support decision-making by the Town representatives. Based on the findings, the town has decided to move forward with state and local permitting of a new groundwater source. Initial permitting has been completed in support of the long-term testing of the proposed site which was completed in fall 2010. Data evaluation and modeling of zones of contribution to the proposed wellfield are ongoing.

MADEP SARSS Program, New Source Approval of a Replacement Municipal Water Supply Source. Holbrook, MA. Project Manager/Field Manager for New Source Approval. The reactivation of the Donna Road aquifer under Operable Unit 4 of the Record of Decision for the Baird and McGuire Superfund site is anticipated to replace the 0.31 million gallons per day of municipal water lost due to the contamination of the South Street Wellfield. Field activities included: extensive surveying of surrounding land uses; installation of numerous exploration, observation, and monitoring wells; geophysical and bedrock fracture trace and fracture fabric analyses; installation of pilot production well(s); discharge water and groundwater sampling; and performance of a long-term aquifer pumping test. Project work also included temporary road design; preparation of draft bylaws and wellhead protection district documents; delineation of the zone of contribution (Zone II) of the proposed supply well; and preparation of documents to satisfy Massachusetts Division of Water Supply guidelines and regulations. Division of Water Supply approval of the source and the Zone II delineation was received in May 1994.

Winchester Country Club. Lake Sediment Assessment in Support of WPA Permitting. Winchester, MA. Task Manager and Hydrogeologist for an evaluation of sediment quality in the Upper Mystic Lake with regard to its potential impact on the quality of groundwater withdrawn from a proposed irrigation well located adjacent to the lake. Mr. Hershberger assisted in the design of the field sampling program, performed analytical modeling of the projected capture zone of the proposed pumping well and coordinated a contaminant fate and transport and risk evaluation of the potential for groundwater withdrawn through sediments of Upper Mystic Lake to mobilize metals contained in the sediments from upstream industrial properties and state- and federally-listed disposal sites. All of the predicted groundwater concentrations were found to be less than the applicable MassDEP standards and other applicable risk thresholds. The predictions have been confirmed by the results of ongoing groundwater sampling from the existing supply well.

Town of Abington, Evaluation of Existing Supply Well and Potential Irrigation Water Demands. Abington, MA. Task Manager and Senior Hydrogeologist for the evaluation of the capacity of the existing supply well and also estimated the water demands to support the potential expansion of the existing irrigation system at the town-owned Strawberry Valley Golf Course. The project evaluated the capacity of the existing water supply system (supply well and irrigation pond) to meet the estimated water demands of the expanded irrigation system.

DARRELL OAKLEY, PWS
Senior Ecologist

Experience
ESS Group, Inc.: 2004 to present

Years of Prior Related Experience: 10

Education
BA, Biology (concentration in Environmental Science), Colby College, 1994

Certificate in Native Plant Studies, New England Wildflower Society, (anticipated completion 2012)

Professional Certifications and Affiliations
OSHA 40 Hour Health & Safety Training for Hazardous Materials Operations

Massachusetts Association of Wetland Scientists

Society of Wetland Scientists, Professional Wetland Scientist (No. 00001424)

State of New Hampshire Wetland Scientist Certification (No. 154)

Qualifications

Mr. Oakley is a Senior Ecologist with a diverse background and 17 years of experience in environmental consulting. He specializes in avian biology; wetland delineation and wetland mitigation; habitat assessment; on-site identification of flora and fauna species; rare, threatened, and endangered species surveys; and endangered species mitigation. Mr. Oakley has completed numerous state and federal environmental applications, including Environmental Impact Statements and state and federal wetland submittals in the New England and mid-Atlantic regions. His project experience includes wind power development, commercial and residential projects, airport site development, local and regional sewer projects, federal government facility siting, railroad projects, gas pipeline installation, power plant siting, fiber optic installation, watershed protection, water diversion, highway and bridge construction, and remedial investigations for hazardous waste sites.

Representative Project Experience

Town of Hopedale – Dredging Feasibility Assessment, Hopedale Pond, Hopedale, MA. Assistant project manager and senior ecologist for an extensive pre-dredging evaluation of Hopedale Pond, a 35 acre mill pond in Hopedale, MA that is suffering the effects of eutrophication and in-filling from its watershed. A goal of the study is to evaluate the quantity and quality of sediment in the pond as well as to assess the nutrient, bacteria, and other water quality issues related to ongoing inputs from its watershed. The results of the study will be used to provide the town with management recommendations for restoring this pond to its former condition through dredging. Management recommendations will include a detailed description of existing sources of pollution from its watershed and conceptual engineering designs for solving these issues on a site-by-site basis. The Best Management Practices (BMPs) that ESS will be recommending will be designed to be economical yet effective. A focus of the ESS strategy will be to implement or retro-fit Low Impact Development (LID) techniques into the existing watershed landscape.

Wannacomet Water Company – Nantucket, MA. The Wannacomet Water Company (Wannacomet) proposed the installation of new wells and a 2,000,000 gallon storage tank on the North Pasture site on the island of Nantucket, Massachusetts. Nantucket has the highest concentration of state-listed rare species in the Commonwealth of Massachusetts. Mr. Oakley was brought in to navigate Wannacomet through the extensive rare species survey requirements and regulatory hurdles. The North Pasture is within Priority Habitat and near Estimated Habitats for 26 state-listed species ([reptile] spotted turtle; [bird] long-eared owl, northern harrier; [moths] chain dot geometer, barrens buckmoth, southern ptichodis, a noctuid moth, barrens daggermoth, straight lined mallow moth, spiny oakworm, pink sallow, coastal swamp metarranthis moth, pine sallow, coastal swamp metarranthis moth, pine barrens zale, Melsheimeri sack bearer, coastal heathland cutworm, and Gerhards's underwing moth; [plants] broom crowberry, Nantucket shadbush, eastern silvery aster, Mattamuskeet panic-grass, St. Andrews cross, New England blazing star, sandplain blue eyed grass, lion's foot, and bushy rockrose). Mr. Oakley conducted extensive surveys on the North Pasture over two years for plants, moths, and birds, in order to document rare species on the site. He conducted daytime and overnight moth surveys, plant surveys during important flowering periods, and searches for nesting Northern Harriers and Long-eared Owls.

Massachusetts Department of Conservation and Recreation – Dredging Design and Permitting, Robinson Pond, Agawam, MA. Assistant project manager and senior ecologist responsible for the restoration of Robinson Pond in Robinson State Park. Mr. Oakley is overseeing the engineering design and permitting for the pond's restoration which includes dredging.

Walpole Country Club – Walpole, MA. Provided support to the Walpole Country Club under the Wetlands Protection Act to manage invasive species and vegetation around the Club's pond. Supported the Country Club in hearings with the Conservation Commission and facilitated permitting. Mr. Oakley designed and oversaw construction of stream bank stabilization projects and selective cuttings in enhancing the vegetation around the pond.

Massachusetts Department of Conservation and Recreation – Dredging Design and Permitting, Farm Pond, Carlisle, MA. Assistant project manager and senior ecologist responsible for the restoration of Farm Pond in Great Neck State Park. Mr. Oakley is overseeing the engineering design and permitting for the pond's restoration which includes dredging. Sediment from the pond will be re-used on the state park property as a landscape amendment.

Town of Norton – Diagnostic and Feasibility Assessment for Management of Lake Winnecunnet, Norton, MA. Contributed to the assessment of Lake Winnecunnet and its watershed. The lake is part of a Massachusetts ACEC (Area of Critical Environmental Concern). The deep-water habitat associated with the lake is threatened by the invasive and exotic plant *Cabomba caroliniana* (fanwort), which has spread throughout the lake to the detriment of native plants and potentially native fauna. The need to manage this situation while protecting the potentially rare or threatened species that exist within the lake required extensive survey of the lake shoreline, the major tributaries to the lake (Canoe River and Mulberry Meadow Brook), and the lake outlet (Snake River). Mr. Oakley surveyed the adjoining wetlands, open water habitats, and shoreline to determine baseline conditions. Mr. Oakley also surveyed the lake and adjoining habitats for rare species, which could be affected by lake management. The results of the survey were used to help develop a comprehensive lake and watershed management plan for the Town.

Department of Conservation and Recreation – Ponkapoag Bog Ecological Monitoring, Canton, MA. Ecologist responsible for monitoring water levels in Ponkapoag Pond and Bog in compliance with an Order of Conditions and Water Level Monitoring Plan issued by the Canton Conservation Commission. These efforts are conducted to preserve the fragile ecosystem of an Atlantic white cedar/emergent/scrub-shrub wetland. Monitor water level and groundwater levels of the pond and bog, and measure discharge rates at Ponkapoag Brook. The study also includes annual assessments of seven vegetation plots, monitoring of Atlantic white cedar growth rates, characterization and mapping of cover types, soils analysis, and assessment of the populations of five state-listed insect species.

Northeast Utilities – Rare Species Habitat Assessment for 38-mile Electric Transmission Line, Lebanon to Chaplin, CT. Assessed potential electric transmission line impacts to two rare habitats and thirteen listed species. Significant Habitats included poor fen and an Atlantic White Cedar Swamp. Rare species included American Bittern, Pied-billed Grebe, Blue-winged Teal, Purple Martin, Northern Saw-wet Owl, Savannah Sparrow, Whip-poor Will, wood turtle (*Glyptemys insculpta*) eastern hognose snake (*Heterodon platyrhinos*), banded bog skimmer (*Williamsonia lintneri*), bog copper (*Lycaena epixanthe*), an aquatic snail (*Gyraulus circumstriatus*) American rubyspot (*Hetaerina americana*) Henry's elfin (*Callophrys henrici*), frosted elfin (*Callophrys irus*), a noctuid moth (*Lepipolys perscripta*) and mustached clubtail (*Gomphus adelphus*). Identified rare species habitats and host plant species for moths. Developed survey protocol to survey for two host plants species: blue toadflax (*Linaria canadensis*) and lupine (*Lupinus perennis*). Worked with Connecticut Department of Environmental Protection to assess and minimize potential impacts.

DANIEL J. HERZLINGER, PWS
Environmental Scientist

Experience
ESS Group, Inc.: 2006 to present

Years of Prior Related Experience: 3

Education
MEM, Resource Ecology, Duke University, 2001

BA, Biology, Bates College, 1997

Professional Registrations and Affiliations
Society of Wetland Scientists - Professional Wetland Scientist

Member of Society of Wetland Scientists

Association of Massachusetts Wetland Scientists – Full Voting Member

40-Hour OSHA Hazwoper Training and 8-Hour Refresher Training (last issued 1/17/2012)

10-Hour OSHA Construction Safety and Health Training

Qualifications

Mr. Herzlinger is a Professional Wetland Scientist (PWS) with over seven years of diverse experience and specialized skills in ecological field investigations, wetland delineations, environmental permit preparation, natural resource site assessments, watershed management plan development, wildlife habitat evaluations and rare species surveys. His range of project experience includes the siting and permitting of energy generation facilities and infrastructure, commercial development, stormwater remediation design, lake management plans and watershed assessments. Mr. Herzlinger has conducted field studies and prepared technical environmental permits for a variety of projects throughout New England and the mid-Atlantic region. He has expertise in a range of environmental regulations, especially in Massachusetts and Rhode Island and regularly represents clients at a variety of regulatory hearings. Mr. Herzlinger has extensive technical expertise in the use of Geographic Information Systems (GIS) and Global Positioning Systems (GPS).

Representative Project Experience

Department of Conservation and Recreation – Flowering Pond Restoration Plan, Newburyport, MA. Assessed the existing ecological value of Flowering Pond and adjacent habitats as part of the development of a pond restoration plan. Implemented a bathymetric survey, isopach (sediment-depth) survey, aquatic plant mapping and sediment sample collection. Assisting with the preparation of the restoration plan.

Town of Concord – Warner's Pond Watershed Management Plan, Concord, MA. Assistant project manager on a project to develop a watershed management plan for Warner's Pond on behalf of the Town of Concord. Coordinated the implementation of the field data collection program which included bathymetric survey, sediment sampling, water quality sampling, watershed assessment and aquatic macrophyte mapping. The results of the field investigations were used to prepare the Watershed Management Plan and provide recommendations for addressing water quality issues in Warner's Pond.

Massachusetts Department of Conservation and Recreation – Pond Dredging, Agawam and Carlisle, MA. Delineated jurisdictional freshwater wetland resource areas under the Massachusetts Wetlands Protection Act at Robinson Pond, in Robinson State Park. Prepared and filed Environmental Notification Forms and Notices of Intent for the proposed dredging of Robinson Pond as well as Farm Pond. Presented projects to regulators at MEPA site visits and local Conservation Commission hearings. Prepared the Request for 401 Water Quality Certifications to the MassDEP and the Section 404 U.S. Army Corps of Engineers applications for each pond. Dredging was conducted to restore aquatic habitat and water quality within the ponds. Responsible for environmental inspection and oversight during the second phase of dredging. prepared and filed environmental compliance documents with state and local regulatory agencies at the completion of the project.

Town of Hopedale – Diagnostic/Feasibility Study of Hopedale Pond, Hopedale, MA. Conducted bathymetry, plant mapping and a sediment depth survey of Hopedale Pond as a component of a study to address water quality and invasive species issues within the pond. Collected dry and wet weather surface water samples within the pond and at strategic locations within the watershed. Prepared sections of a

diagnostic/feasibility study including background research and biological controls to manage invasive species.

Town of Stoughton – Ames Long Pond Plant Management, Stoughton, MA. Ames Long Pond has been impacted by dense growth of invasive, aquatic species. A pilot hydro-raking program was implemented to test the program's cost-effectiveness at removing invasive aquatic species. Conducted a pre and post-hydroraking plant survey and prepared a letter report outlining the overall success of the program, with recommendations for future plant management at Ames Long Pond.

West Brookfield Stormwater Authority – Lake Wickaboag Stormwater Improvement Project, West Brookfield, MA. Completed a wetland delineation and wildlife habitat assessment for this proposed dredging and wetland creation project. Filed the Environmental Notification Form for the project and prepared the Notice of Intent under the Massachusetts Wetlands Protection Act. Representing the client at regulatory hearings on the project.

Town of Sharon – Hydrogeologic Investigation for Source Water Development, Sharon, MA. The Town of Sharon is seeking to develop a new drinking water source from an aquifer in town and install wells to conduct the initial pump tests. Delineated wetland resource areas under the Massachusetts Wetlands Protection Act at the site and prepared the Notice of Intent for the proposed well installation and pump tests. Evaluated the soils, hydrology and vegetation of wetlands at the site to determine the potential impact that groundwater withdrawal will have on these wetland resource areas.

Walpole Country Club – Regulatory Permitting and Engineering Design Services, Allen Pond, Walpole, MA. Delineated wetland resource areas adjacent to a 3.5-acre pond in accordance with the Massachusetts Wetlands Protection Act and U.S. Army Corps of Engineers, Wetland Delineation Manual (1987) for dredge work. Completed a Wildlife Habitat Evaluation conducted in accordance with Appendix B of the *Massachusetts Wildlife Habitat Protection Guidelines for Inland Wetlands*. Prepared the Notice of Intent and MEPA review applications for proposed dredge work at Allen Pond. Prepared the Request for 401 Water Quality Certification and Section 404 Army Corps application for the project.

Plymouth EDF – Rare Species and Habitat Mapping, Plymouth, MA. Completed a survey of a 1,000-acre parcel to assess natural communities at the site and evaluate constraints on development based on the presence of rare natural communities and species. Mapped the location of rare natural communities and produced GIS figures delineating sensitive areas based on the field assessment.

Housatonic River Natural Resource Damage Fund – Housatonic River Enhanced Public Access Project, Housatonic River, MA. Partnered with the Housatonic Valley Association to perform an initial screening of 40 potential sites for enhanced public access to the Housatonic River in western Massachusetts. The screening was based on land availability as well as physical, hydrological, and natural resource constraints. Conducted rare species surveys, evaluated access constraints and collected data on stream profiles, streambed composition and substrate characteristics. Conducted field surveys for the presence of Jefferson and Four-toed Salamanders, which are listed as species of special concern in Massachusetts.Delineated jurisdictional wetland resource areas at five high priority sites and prepared the Notices of Intent under the Massachusetts Wetlands Protection Act for construction of canoe launches at each of these five sites. Presented the project before the Conservation Commissions in the five towns with proposed canoe launch sites and successfully permitted all of the launches. Construction was completed in 2010.

MATTHEW D. LADEWIG, CLM
Aquatic Ecologist

Experience
ESS Group, Inc.: 2006 to present

Years of Prior Related Experience: 3

Education
MS, Aquatic Resource Ecology and Management, University of Michigan, 2006

BA, Geography, University of Illinois at Urbana-Champaign, 2000

Professional Registrations and Affiliations
North American Lake Management Society: Certified Lake Manager

Society for Freshwater Science: Certified Taxonomist for Eastern North America

Rhode Island Natural History Survey

Service
Ten Mile River Watershed Council Annual Herring Scoop – River Herring Handler (2008 to 2010)

Rhode Island Natural History Survey Annual BioBlitz – Aquatic Insect Taxonomist (2007 to Present)

Qualifications

Mr. Ladewig is an ecologist with experience in the monitoring, modeling, and management of aquatic ecosystems. He is directly involved in field work, laboratory work, data analysis, modeling, reporting, and presentation phases of lake management projects and has studied over 40 lakes and ponds to date. Mr. Ladewig is proficient in the taxonomy of a wide variety of aquatic and terrestrial organisms and is a certified aquatic macroinvertebrate taxonomist through the Society for Freshwater Science. He conducts rare species surveys for several aquatic species, including freshwater mussels, dragonflies, and damselflies. These skills are complemented by an understanding of aquatic physicochemical processes and knowledge of lake management tools. Additionally, Mr. Ladewig possesses a wide variety of analytical skills, including the operation of statistical software for ecological data analysis and Geographic Information System (GIS) software for spatial analysis and mapping.

Representative Project Experience

Massachusetts Department of Conservation and Recreation – Lakes and Ponds Program, Restoration of Flowering Pond, Newburyport, MA. Analyzed the results of the algae sampling program and conducted a hydrologic analysis for pond drawdown. Provided a dredge feasibility analyses for Flowering Pond based on sediment isopach mapping and sediment physicochemical characterizations conducted as part of the project. The goal of the project is to restore the aesthetic and recreational attributes of Flowering Pond while maintaining its ecological value.

Brooks Pond Conservation Association – Development of a Lake Management Plan, North Brookfield, New Braintree, Oakham, and Spencer, MA. Led field program at Brooks Pond, including water quality sampling and aquatic macrophyte mapping. Developed a lake management plan with short and long term recommendations for maintaining the recreational and ecological assets of the pond. Assisted client and Town of North Brookfield with submittal of a proposal for grant funding under Section 319 of the Clean Water Act.

Mill Pond Committee – Development of a Pond Restoration Plan for Mill Pond, West Tisbury, MA. Evaluated the economic and technical feasibility of various lake management options for the cost-effective restoration of Mill Pond, a small pond that is currently used for passive recreation and fishing. Recommended management actions for restoration of the pond included dredging of the southern basin to restore water volume and reduce a significant source of nutrients (internal recycling) as well as creation of a treatment wetland at the pond inlet to sequester nutrients sourced from the watershed.

Massachusetts Water Resources Authority – Aquatic Invasive Macrophyte Surveys, MA. Managed field effort and reporting tasks for a comprehensive survey of aquatic macrophytes at ten source and emergency reservoir areas jointly managed by MWRA and the Massachusetts Department of Conservation and Recreation. This survey provided the first comprehensive update to baseline macrophyte surveys completed in 2006 and 2007. Developed aquatic macrophyte monitoring and management plan that included an assessment of climate change impacts on macrophyte communities in the MWRA/DCR reservoirs. Compiled the first comprehensive field guide to the aquatic macrophytes of the entire MWRA/DCR reservoir system.

Quaboag and Quacumquasit Lake Association – Aquatic Invasive Weed Control Pilot Study, Lake Quacumquasit, East Brookfield, Brookfield, and Sturbridge, MA. Conducts pilot study of cost-effective, small scale treatments for control of invasive aquatic weeds. In response to persistent invasive weed problems at Quaboag Pond and Lake Quacumquasit, ESS developed a long-term plant management plan. As an initial management step, a pilot study will be conducted to investigate the success of several low-cost alternatives to lakewide herbicide treatment. Experimental treatments will be feasibility tested in aquaria trials. Treatments with the greatest likelihood for success will be studied in-situ using enclosures to isolate dense weed beds of invasive Eurasian milfoil (*Myriophyllum spicatum*) and fanwort (*Cabomba caroliniana*). Field testing will allow ESS to provide QQLA with a toolbox of small-scale management actions that can be used to control weed growth in key recreational areas at minimal cost.

Town of Wellesley – Phytoplankton and Water Quality Monitoring of Morses Pond, Wellesley, MA. Conducted monitoring of in-lake conditions at Morses Pond, a 103-acre lake within a highly urbanized setting. Responsibilities included water quality sampling and collecting Secchi disk readings and phytoplankton samples. Also provided rapid turnaround screening level identification of phytoplankton samples to detect incipient algae blooms that could impact recreational use at the pond. Analyzed water quality and phytoplankton data for final reporting.

Town of Hopedale – Diagnostic/Feasibility Study of Hopedale Pond, Hopedale, MA. Led seepage survey of Hopedale Pond shoreline to evaluate potential groundwater sources of bacteria and nutrients. Also assisted with collection of dry weather surface water samples within the pond and at strategic locations within the watershed. Drafted several sections of the diagnostic/feasibility study report, such as management recommendations to control Canada goose overpopulation and associated nutrient pollution, fecal contamination, and general incompatibility with public uses at the pond. Additionally, developed a Canada goose pilot study to assess and evaluate the success of management options as they are adopted by the town.

Massachusetts Department of Conservation and Recreation – Lakes and Ponds Program, Sampling, Design and Permitting Services to Support Dredging at Robinson Pond and Farm Pond, Agawam and Carlisle, MA. Assisted with the design and implementation of a sediment sampling plan for two small ponds on state-managed land. The principal objectives of this project were to assist the client in obtaining the necessary environmental permits for dredging and onsite disposal as well as prepare the final engineering drawings for each pond.

Town of Westford – Baseline Characterization, Drawdown Feasibility Assessment, and Long-term Monitoring Program for Nabnasset Lake, Westford, MA. Conducts aquatic plant surveys and monitors macroinvertebrates and water quality in Nabnasset Lake. Monitoring is required by an Order of Conditions to evaluate the impacts of annual winter lake drawdowns for the purpose of controlling nuisance aquatic plants.

Massachusetts Department of Conservation and Recreation – Ponkapoag Golf Course, Water Supply Development and Ecological Monitoring, Canton, MA. Conducts biological surveys for several state-listed butterflies, damselflies and dragonflies. Monitors water levels in Ponkapoag Pond and Bog in compliance with an Order of Conditions and Water Level Monitoring Plan issued by the Canton Conservation Commission. These efforts are conducted to preserve the fragile ecosystem of an Atlantic white cedar/emergent/scrub-shrub wetland.

Gomez and Sullivan – Housatonic River Freshwater Mussel Survey, Glendale Power Station, Stockbridge, MA. Assisted with a field survey for mussels in the bypass channel of a hydro power station on the Housatonic River. In addition, was responsible for filing a Rare Animal Observation Form with the Massachusetts Natural Heritage and Endangered Species Program when evidence of a state-listed mussel species was found in the channel. Summarized the findings of the survey in a report to the client for compliance with Federal Energy Regulatory Commission (FERC) relicensing procedures.

JANET CARTER BERNARDO, PE
Senior Civil Engineer

Experience
ESS Group, Inc.: 1997 to present

Years of Prior Related Experience: 8

Education
BS, Civil Engineering, University of Lowell, 1984

Numerous professional seminars and conferences

Professional Registrations
Massachusetts Registered Professional Engineer - No. 34700

Massachusetts Soil Evaluator – No. 148

Massachusetts System Inspector (Septic) – No. 1887

New Hampshire Subsurface Disposal Designer - No. 1340

New Hampshire Registered Professional Engineer – No. 11865

New York Registered Professional Engineer – No. 078701-1

Virginia Registered Professional Engineer – No. 048864

Qualifications

Ms. Bernardo is a registered Professional Civil Engineer (PE) with technical and management experience in civil site design. As a Project Engineer and Manager, Ms. Bernardo has managed and participated in numerous site designs and permitting projects, including industrial, office, retail, commercial, and residential properties. These projects include zoning analysis, building and parking layouts, traffic analysis, stormwater management, drainage and utility design, subsurface disposal system design, construction details, specifications and construction administration. She is also experienced in local and state permitting and has served as the reviewing consultant for various Massachusetts communities. Ms. Bernardo is currently a board member on the Needham Conservation Commission and the Needham Community Preservation Committee.

Representative Project Experience

Stormwater Authority, Wickaboag Valley Road. West Brookfield, MA. Senior Civil Engineer responsible for designing a stormwater management system to improve the runoff characteristics of stormwater prior to it discharging to Lake Wickaboag. Assisted the West Brookfield Stormwater Authority with design options, construction drawings, cost estimate, and construction oversight for the preferred stormwater management system.

Village Landing Park, Canoe Launch. Everett, MA. Senior Engineer responsible for design oversight to facilitate construction of a canoe and kayak launch at this former industrial site located on the banks of the Malden River, which has been re-developed into a public park by the City of Everett. The canoe and kayak launch will enhance public access to and use of the Malden River for recreational activities.

Town of Hopkinton, Stormwater Drainage Analysis. Hopkinton, MA. Project Manager and Senior Civil Engineer responsible for the data research, drainage analysis, design recommendations, and Subdivision Regulation revisions for the town's Planning Board in regard to the White Oak Estates subdivision, Spring Street, and the Whitehall Reservoir.

Local Planning Boards and Conservation Commissions, Peer Review Services. Groton, Pepperell, Dartmouth, and Burlington, MA. Peer Review Consultant responsible for reviewing numerous site plans, stormwater management, subdivisions, and septic designs presented to the local Planning Boards and/or Conservation Commissions by other civil engineering firms. Responsibilities include making recommendations to the approving Board on the accuracy of the submitted design in accordance with the state and local regulations as well as good engineering practice.

Town of Andover, Stormwater Peer Review. Andover, MA. Senior Civil Engineer providing ongoing peer review services to the Andover Planning Board and the Andover Conservation Commission. Responsibilities include evaluating the adequacy and appropriateness of the proposed stormwater management design in accordance with the local wetlands and stormwater bylaws, MassDEP Stormwater Management Guidelines, and sound engineering practice.

Winchester Country Club, Short Range Course and Practice Facility. Winchester, MA. Senior Engineer responsible for the layout, utility connections, and stormwater management plan for the proposed short-range course and practice facility with clubhouse building and accessory areas. Permitting involves submitting a Notice of Intent to the Winchester Conservation Commission.

Pembroke Real Estate, Boston Coach. Everett, MA. Project Manager for site design and permitting of a 250-space parking lot with extensive stormwater infiltration system. Design included parking and access roadway configuration, a handicap accessible ramp, grading, drainage, and landscaping. Permits involved Modification of an Order of Conditions and an MWRA 8(m) permit. Responsibilities included preparation of a SWPPP and construction oversight.

Berkeley Green II, LLC, RiverGreen Technology Park, Everett, MA. Senior Engineer responsible for the civil/site design of a state-of-the-art technology park focused on research and development for renewable energy, green technology, and biotechnology. The RiverGreen project will consist of approximately 500,000 sf of flexible space on a 40-acre former Brownfield site along the Malden River. ESS is conducting geotechnical and geophysical site investigation; managing all environmental issues related to previous site contamination; civil site design and all required permitting. Stormwater management was designed utilizing numerous best management practices in accordance with MassDEP's Stormwater Management Policy. Permitting of the project includes obtaining an Order of Conditions from the Everett Conservation Commission, Subdivision Approval from the Everett Planning Board, Ch91 licensing, Massachusetts Environmental Policy Act, and an MWRA 8(m) permit. A Stormwater Pollution Prevention Plan (SWPPP) was prepared for roadway construction and the earthwork activities.

W/S Development Associates LLC, Retail Shopping Center. Wareham, MA. Engineer of Record responsible for reviewing site layout, grading, drainage, and utility design for a 750,000 square foot retail and restaurant community style open air shopping center located at the interchange of Route 28 and Interstate 195. Work included conceptual planning, access design, parking facility design, grading and earthworks, stormwater management, and utility infrastructure design. Oversaw administration for construction phase services including shop drawing review, responses to requests for information, review of payment requisitions, and submissions of field change bulletins.

Bentley College, Residence Halls. Waltham, MA. Engineer of Record responsible for reviewing site layout, grading, drainage, and utility design for the development of two, three-story residence dormitories for the housing of 120 students on campus. Work included the design of additional parking fields and access roadways, stormwater management systems, utility infrastructure, and site grading and layout.

Winchester Country Club, Maintenance Facility. Winchester, MA. Senior Engineer responsible for the layout, utility connections, and stormwater management plan for the maintenance facility buildings and accessory areas. Permitting involved Conservation Commission Order of Conditions.

W/S Development Associates LLC, Retail Shopping Center. Mansfield, MA. Senior Civil Engineer responsible for reviewing site layout, grading, drainage, and utility design for a 395,000 square foot retail and restaurant community style open air shopping center located at the interchange of Route 140 and Interstate 495. Work included conceptual planning, access design, parking facility design, grading and earthworks, stormwater management, and utility infrastructure design. Oversaw administration for construction phase services including shop drawing review, responses to requests for information, review of payment requisitions, and submissions of field change bulletins.

ERICA URIARTE, EIT
Civil Engineer

Experience
ESS Group, Inc.: 2006 to present

Years of Prior Related Experience: 1

Education
BS, Civil & Environmental Engineering, Clarkson University, 2004

Professional Registrations
Registered Engineer-in-Training, Massachusetts

Affiliations
American Society of Civil Engineers

Qualifications

Ms. Uriarte has six years of experience related to civil/site development of residential subdivisions and commercial properties. Her civil work includes site grading, earthwork analysis, storm water management system design, utility research and layout, dredging, and subsurface disposal system design. Ms. Uriarte has assisted in the preparation of various environmental regulatory permit applications including Chapter 91 Waterways License applications, 401 Water Quality Certifications, Notices of Intent, and New York Article VII submittals. Ms. Uriarte's experience also includes preparing construction specifications, reviewing shop drawings, and construction oversight. She also has experience in the planning and permitting of linear transmission projects, including preparation of desktop routing studies. In addition, she has technical experience with computer programs including AutoDesk Land Desktop, Hydraflow, and HydroCAD.

Representative Project Experience

West Brookfield Stormwater Authority – 319 Non-point Source Pollution Grant, Lake Wickaboag Stormwater Improvements Project, West Brookfield, MA. Designed a treatment train of sediment forebays and constructed stormwater wetlands within existing drainage basins located upstream of Lake Wickaboag to improve the water quality of stormwater runoff discharging to the lake. Responsible for the design of outlet control structures for seasonal drawdown and for future operation and maintenance of the basins. Work includes providing detailed site plans for various permit submittals, attending monthly meetings with the West Brookfield Stormwater Authority, and providing construction documents and costing.

Scarfo Construction, LLC - Lots 56R-105 and 56R-47, Westfield, MA. Staff Engineer assisting in the design of a Site Stabilization Plan and Bordering Vegetated Wetland (BVW) Replacement Plans for the restoration of a BVW and stabilization of a stream per the Massachusetts River and Stream Crossing Standards and Massachusetts Stream Crossing Handbook. Prepared detailed AutoCAD plans of the design for submission to Massachusetts Department of Environmental Protection (MassDEP) and Natural Heritage.

West Brookfield Stormwater Authority – West Brookfield, MA. Staff Engineer responsible for designing a stormwater management system to improve the water quality of the stormwater runoff discharging to Lake Wickaboag from Wickaboag Valley Road. The stormwater analysis and design included the use of LID practices to reduce the phosphorus loading to the lake. Submitted a NOI to the West Brookfield Conservation Commission and presented the project to the commission during a public hearing. Also conducted construction over site during the installation of the system.

Massachusetts Department of Conservation and Recreation (DCR) — DCR Pond Dredging, Agawam, MA. Responsible for the dredging and dewatering design of Robinson Pond in Robinson State Park. Work included preparing the site plans necessary to support the 401 Water Quality Certification to MassDEP and the Section 404 Army Corps application, writing a Construction SWPPP, as well as conducting construction oversight to ensure dredging operations were being performed in accordance with the approved drawings and specifications.

Walpole Country Club – Dredging Feasibility Assessment for Allen Pond, Walpole, MA. Responsible for the dredging and dewatering design as well as erosion and sediment control of sediment

removal from Allen Pond on the Walpole Country Club property. Work includes providing detailed AutoCAD plans for the pond restoration. The ultimate goal of the project is to restore the ecological and aesthetic value of the pond.

Moskowitz – Pond Dredging, Belmont, MA. Responsible for the dredging and dewatering design as well as erosion and sediment control of sediment removal from the pond located in the backyard of a residential property. Work included providing detailed AutoCAD plans for the submission of a Notice of Intent to the local conservation commission and providing construction oversight to ensure the dredging operations were being performed in accordance with the approved drawings.

Littleton Water Department – Public Access to Mill Pond, Littleton, MA. Staff Engineer responsible for the design of a canoe launch and fishing platform at Lake Warren Street on the bank of Mill Pond. Also Responsible for designing a parking area and pervious paver walkway to the launch site. The purpose of the launch is to enhance public access to the pond for recreational activities.

City of Everett – Public River Access Development, Malden River, Everett, MA. Staff Engineer responsible for the design of a canoe and kayak launch at the City's Village Landing Park – an underutilized green space created on a capped landfill located on the banks of the Malden River. The purpose of the canoe and kayak launch is to enhance public access to the Malden River for recreational activities.

Town of Andover – Stormwater Technical Peer Review, Andover, MA. Responsible for evaluating stormwater management designs for multiple projects as part of the technical peer review services ESS provides to the Andover Planning Board and the Andover Conservation Commission. Determined whether designs submitted by various applicants are in accordance with the local regulations and MassDEP Storm Water Management Guidelines.

Town of Brookfield – 319 Non-point Source Pollution Grant, Brookfield, East Brookfield and Spencer, MA. Conducted a field assessment to determine possible sources of phosphorous loading within the Quaboag Pond and Quacumquasit Pond watersheds. The results of the assessment and monitoring were used to research, design, and implement Best Management Practices (BMPs) to address the non-point source pollution.

TD Banknorth – Wetland Permitting, Concord, MA. Staff Engineer responsible for the design of stormwater management improvements as part of a Notice of Intent submission to bring Banknorth's facilities into compliance with the Americans with Disabilities Act (ADA) regulations (28 CFR Part 36) through the installation of a wheelchair accessible ramp. The project was located within the floodplain of the Concord River, and required design of compensatory flood storage to mitigate for the loss of flood storage volume associated with the site improvements. Prepared compensatory flood storage calculations to support the NOI application and generated AutoCAD plans.

Berkeley Green II, LLC. – RiverGreen Business Park, Everett, MA. Staff Engineer involved in the civil/site design of a multi-use industrial, office, research and development center. Wrote the Stormwater Management Report for the Expanded Environmental Notification Form (EENF), Notice of Intent (NOI), Single Environmental Impact Report (SEIR), and the Definitive Subdivision. Incorporated Low Impact Development (LID) techniques into the stormwater management design by using gravel wetlands, bioretention areas, wet basins, and grassed swales as well as provided stormwater analysis of these systems. Generated utility layout and design. Organized and attended meetings with the City of Everett and Massachusetts Water Resources Authority (MWRA). Responsibilities also included generating Phase 1 Construction Drawings, preparing a Construction Stormwater Pollution Prevention Plan (SWPPP), and conducting construction oversight. Construction Oversight included reviewing shop drawings, responding to Requests for Information (RFIs), and conducting field visits.

Troy Daryl Tuckey

Virginia Institute of Marine Science
College of William and Mary
PO Box 1346
Gloucester Point, VA 23062
804-684-7328
tuckey@vims.edu

Education:

2004 – 2009	Ph.D. Fisheries Science Virginia Institute of Marine Science College of William and Mary Major advisor: John E. Olney
1996-2000	Master of Science in Marine Biology University of Charleston Major Advisor: John C. McGovern
1989 – 1993	Bachelor of Science in Biology University of South Florida

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Professional Goals and interests:

Perform scientific research related to fisheries population dynamics. Apply research results to pertinent problems and serve an advisory role to stakeholder groups. Collaborate locally and abroad to produce the best possible science and learn or develop new techniques. Mentor students and actively teach fundamentals of fishery science as well as advanced tools and techniques.

Professional Experience:

June 2008 - Marine Scientist II (Supervisor)
Program manager
Juvenile Fish and Blue Crab Trawl Survey
Virginia Institute of Marine Science

Duties: Supervise and manage all aspects of the juvenile fish and blue crab trawl survey. Includes scientific oversight, budgetary oversight, proposal writing, personnel management, advisory service and outreach. Oversee two survey's examining recruitment of glass American eel in the Potomac River and Virginia.

Apr 2003 - Jul 2004 Research Administrator I
Florida Fish and Wildlife Conservation Commission, Florida Marine Research Institute, Apalachicola Field Laboratory, Eastpoint, FL

Duties: Supervised and participated in operations of a field laboratory that monitored abundance of estuarine fish. Developed and conducted scientifically sound research projects that addressed current needs or interest. Supervised, and trained staff. Maintained field sampling equipment including boats, vehicles and trailers, as well as laboratory equipment. Performed budget oversight duties and coordinated with six other field stations to maximize productivity. Participated in regional issues that involved marine concerns and performed public outreach and education.

Sep 2001 - Apr 2003 Assistant Research Scientist
Florida Fish and Wildlife Conservation Commission, Florida Marine Research Institute, Apalachicola Field Laboratory, Eastpoint, FL

Duties: Supervised and participated in operations of a field laboratory that monitored abundance of estuarine fish. Developed and conducted scientifically sound research projects that addressed current needs or interest. Supervised, and trained staff. Maintained field sampling equipment including boats, vehicles and trailers, as well as laboratory equipment. Performed budget oversight duties and coordinated with six other field stations to maximize productivity. Participated in regional issues that involved marine concerns and performed public outreach and education.

Nov 1999 – Sep 2001 Marine Research Associate
Fish & Wildlife Conservation Commission,
Florida Marine Research Institute, Cedar Key Field Laboratory

Duties: Collection and identification of fishes for fisheries independent monitoring program. Maintained database for Cedar Key facility as well as maintained other necessary sampling equipment. Supervised hourly employees and trained new hires on field and laboratory procedures. Developed and conducted scientifically based research projects focusing on areas of special need or current interest.

Mar 1999-Nov 1999 Research Assistant
South Carolina Department of Natural Resources

Duties: South Carolina blue crab stock assessment – Collected and measured blues crabs (*Callinectes sapidus*) for fishery independent survey. Combined blue crab data and physical data obtained from local agencies into Microsoft Access database.

May 1997-Apr 1999 Research Assistant
South Carolina Department of Natural Resources

Duties: Decapod transport study – Responsible for field collection, sorting and identification of postlarval penaeid shrimp.

Aug 1996-Apr 1997 Teaching Assistant
College of Charleston
General biology laboratory instructor.

Duties: Responsible for developing introductory lecture material and examinations.

Dec 1993-Aug 1996 Staff Biologist/ Taxonomist
Mote Marine Laboratory, Benthic Ecology Section

Duties: Assisted in the collection, processing and analysis of benthic invertebrate samples. Responsible for the identification of annelids, specifically freshwater, marine and estuarine polychaetes and oligochaetes. Supervised technicians and instructed junior taxonomists in annelid identification and standard laboratory procedures. Conducted over 50 scientific dives including sediment core collection, moored instrument cleaning, hard bottom delineation, seagrass and reef surveys.

Aug 1993 Technician
Mote Marine Laboratory, Benthic Ecology Section

Duties: Assisted in the collection and sorting of benthic samples.

Presentations:

Tuckey, T. D. and M. C. Fabrizio. 2011. Potential changes in fish relative abundance and community structure in the York River associated with the expansion of blue catfish populations. York River research Symposium, Gloucester Point, VA.

Tuckey, T. D. and M. C. Fabrizio. 2011. Influence of survey design on juvenile fish community metrics: Implications from a study in Chesapeake Bay tributaries. 25th Annual meeting of the Tidewater Chapter of the American Fisheries Society, Gloucester Point, VA.

Brooks, G. H, T. Munroe, and T. D. Tuckey. 2010. Multi-decadal Abundance of Hogchokers (Achiridae: *Trinectes maculatus*) in Lower Chesapeake Bay and Its Tributaries. 25th Annual meeting of the Tidewater Chapter of the American Fisheries Society, Gloucester Point, VA.

Brooks, G. H, T. Munroe, and T. D. Tuckey. 2010. Multi-decadal Abundance of Hogchokers (Achiridae: *Trinectes maculatus*) in Lower Chesapeake Bay and Its Tributaries. 12th Flatfish Biology Conference, Westbrook, CT.

Tuckey, T. D. and M. C. Fabrizo. 2010. Influence of Survey Design on Juvenile Fish Community Metrics: Implications from a Study in Chesapeake Bay Tributaries Annual meeting of the American Fisheries Society, Pittsburg, PA.

Latour, R. J., E. J. Hilton, B. E. Watkins, T. D. Tuckey, P. D. Lynch, and J.E. Olney. 2010. Evaluating restoration efforts of American shad in Virginia. Annual meeting of the American Fisheries Society, Pittsburg, PA.

Tuckey, T. D. 2009. Estimating relative abundance of American eel recruitment to the Potomac River. Potomac River Fisheries Commission, Colonial Beach, VA.

Tuckey, T. D. 2009. American eels in the lower portion of Chesapeake Bay. Presentation to the ASMFC American eel Technical Committee and Stock Assessment subcommittee. Annapolis, MD.

Tuckey, T. D. 2008. Estimating relative abundance of American eel recruitment to the Potomac River. Potomac River Fisheries Commission, Colonial Beach, VA.

Tuckey, T. D. 2008. Population dynamics of juvenile American shad and blueback herring in lower Chesapeake Bay nurseries. 138th Annual meeting of the American Fisheries Society, Ottawa, ON.

Tuckey, T. D. and J. E. Olney. 2007. Abundance of juvenile American shad may be used to predict indices of spawning adults. 137th Annual meeting of the American Fisheries Society, San Francisco, CA.

Tuckey, T. D., J. E. Olney, and A. Weaver. 2006. Growth of wild and hatchery-reared juvenile American shad in the Rappahannock River, Virginia. 30th Annual Larval Fish Conference, Lake Placid, N.Y.

Tuckey, T., Yochum, N., Hoenig, J., Lucy, J., Hepworth, D., Cimino, D. 2005. Evaluating local vs regional management: Tautog (Tautoga onitis) in Virginia. Amercian Society of Ichthyologists and Herpetologists, Tampa, FL.

Tuckey, T., L. Edmiston, and G. Lewis. 2003 Apalachicola Bay and the importance of Freshwater flow. Symposium presentation at the 23rd annual meeting of the Florida Chapter of the American Fisheries Society, Brooksville, FL.

Tuckey, T. 2003. The use of a multi-seine sampling approach to track spot (*Leiostomus xanthurus*) cohorts in three Florida estuaries. Poster presentation at the 23rd annual meeting of the Florida Chapter of the American Fisheries Society, Brooksville, FL.

Tuckey, T. and M. DeHaven.2002. Comparison of fish assemblages in tidal creeks and seagrass habitats in the Suwannee River estuary. Presented at the 22nd annual meeting of the Florida Chapter of the American Fisheries Society, Brooksville, FL.

Tuckey, T. 1999. Recruitment and retention of larval and juvenile fishes in the Ogeechee River, GA. Presented at the American Fisheries Society: Early Life History Section Meeting, Beaufort, NC.

Publications

R. J. Latour, E. J. Hilton, P. D. Lynch, B. E. Watkins, T. D. Tuckey, and J. E. Olney. *In press*. Evaluating restoration efforts of American shad (*Alosa sapidissima*) in Virginia. Marine and Coastal Fisheries.

Tuckey, T. D. and J. E. Olney. 2010. Maturity schedules of female American shad vary at small spatial scales in Chesapeake Bay. North American Journal of Fisheries Management 30:1020-1031.

Tuckey, T. D. 2009. Variability in juvenile growth, mortality, maturity, and abundance of American shad and blueback herring in Virginia. PhD Dissertation, College of William & Mary, 195p.

Tuckey, T. D., N. Yochum, J. Hoenig, J. Lucy, and J. Cimino. 2007. Evaluating Localized vs Large-scale Management: The Example of Tautog in Virginia. Fisheries 32(1):21-28.

Tuckey, T. D. and M. Dehaven. 2006. Fish assemblages found in tidal-creek and seagrass habitats in the Suwannee River estuary. Fishery Bulletin 104:102-117

Technical Reports

Ecosystem Based Fisheries Management for Chesapeake Bay. Alosine species team background and issue briefs. 2011. Maryland Sea Grant College Program , 134 p.

Hoenig, J. M. and T. Tuckey. 2007. Analysis of Virginia tautog age composition data through 2006: A report to the Virginia Marine Resources Commission, March 19, 2007.

Hoenig, J. M., J. Lucy, T. Tuckey, N. Yochum, R. O'Reilly, and J. Cimino. 2005. Evaluating Localized vs Large-scale Management for Virginia Tautog (*Tautogaonitis)*. Technical report to ASMFC.

Advisory service:

ASMFC American eel Technical Committee and Stock Assessment Subcommittee
Shad and river herring subcommittee
Potomac River Fisheries Commission - American eel recruitment
MD Sea Grant Ecosystem Based Fisheries Management Alosine Team, Chair and member of the Fisheries Ecosystem Workgroup
Virginia Marine Resources Commission – tautog management
State of Florida 503b requirements, Minimum Flows and Levels
Journal reviews: Marine Ecology Progress Series, Transactions of the American Fisheries, Journal of Applied Ichthyology, Canadian Journal of Fisheries and Aquatic Science

Appendix B

ESS Standard Operating Guidelines

STANDARD OPERATING GUIDELINES FOR THE CREATION OF A BATHYMETRY MAP

1.0 INTRODUCTION

1.1 Purpose and Applicability

This Standard Operating Guideline (SOG) provides basic instructions for the mapping of depth contours within standing waterbodies. The methods outlined below are intended (1) to standardize depth measurement techniques used by ESS Group field personnel; (2) to standardize the recording of depth measurements to ensure the creation of an accurate bathymetry map.

2.0 RESPONSIBILITIES

2.1 Project Manager

The project manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the measurements in accordance with this SOG and the project plan.

2.2 Field Personnel

The field personnel are responsible for taking accurate depth measurements at documented locations throughout the waterbody. The field personnel are also responsible for recording the number of depth measurements that will best characterize the bathymetric contours of the waterbody, i.e. steep contour areas with coves will be more thoroughly characterized than shallow contour areas with no coves.

3.0 REQUIRED MATERIALS

- The following materials are necessary for the creation of a bathymetry map:
- Boat
- Depth Probe
- Measuring Pole 10ft in length. Marked off in 1ft increments
- Enlarged outline of the waterbody on write-in-the-rain paper
- Global Positioning System (GPS) unit (optional)
- Field note book
- Historical bathymetric maps for the waterbody (optional)

4.0 METHOD

4.1 Depth Measurement Procedure

- A number of transects will be drawn on the map of the waterbody to act as a guide in the collection of depth measurements. The number and location of transects selected will depend on the size and shape of the waterbody, with the aim of thoroughly characterizing the bathymetric contours within it. Historical bathymetric maps can be used (if available) to guide in the selection of transect locations so that areas requiring more thorough characterization can be identified.
- The boat will be driven along each transect, at appropriately spaced points along the transect the boat will be stopped and a measure of the depth of the water at that point will be recorded.
- The number of depth measurement points will depend on the rate of change in depth as the boat is moved along each transect, i.e. the steeper the slope of the waterbody bottom, the more depth

measurements will be taken in order to illustrate incremental changes in depth (i.e. 1ft, 2ft or 5ft increments).

- Each depth measurement point along the transect will be numbered and marked onto the map in order to later link depth data with location information. Locations may be estimated based on landmarks and shoreline morphometry or more precisely mapped using a Global Positioning Systems (GPS). The depth at each point will also be noted with its associated transect and point number in the field note book.
- At each measurement point when the depth is 10ft or less, a measuring pole will be used to measure the exact depth of the water in feet. At depths greater than 10ft a sonar depth probe will be used. This approach minimizes the possibility of plant growth interfering with sonar measurements.

4.2 Creation of Bathymetry Maps

- In the office, depth measurements recorded from throughout the waterbody will be linked with the transects and measurement point locations drawn onto the outline map.
- The known depths at known locations throughout the water body will then be used as a guide (or base) for the drawing of contour lines onto the outline map, thus illustrating incremental changes in water depth either in 1ft, 2ft or 5ft increments depending on the overall depth of the water body.

5.0 QUALITY CONTROL

At each depth measurement point, no matter which depth equipment is being used, a couple of measurements will be taken in very close proximity to each other to make sure the readings are the same, in case of rocks, plants, or other obstacles on the bottom are affecting the measurement at one specific point. In instances where the the measurements are slightly different, the average depth will be recorded.

6.0 DOCUMENTATION

Depth measurements will be recorded in field note books associated with location information in the form of transect numbers and depth measurements points, by ESS personnel. The locations of transect lines and depth measurement points will be recorded on a write-in-the-rain map outline of the waterbody. Any unanticipated site specific information, which requires ESS field personnel to deviate from the above SOG will be reported in an ESS field notebook. Documentation for recorded data must include a minimum of the following:

- Date of survey
- Weather conditions
- Signature or initials of person performing the survey
- Depth measurement point locations
- Comments/Observations

7.0 TRAINING/QUALIFICATIONS

To properly complete an assessment of depth contours within a waterbody, the analyst must be familiar with the measurement and data collection protocols as stated within this SOG and must have confidence in the use of depth measurement equipment.

STANDARD OPERATING GUIDELINESFOR COLLECTION OF SEDIMENTS FROM FRESHWATER ENVIRONMENTS

1.0 INTRODUCTION

1.1 Purpose and Applicability

These Standard Operating Guidelines (SOGs) provide basic instructions for the collection of bottom sediments from freshwater environments. Collections are to be performed in accordance with methodologies generally accepted by the Massachusetts Department of Environmental Protection (MADEP). Laboratory analysis of sediment samples should be performed by a state certified laboratory with the detection limits for analysis specified on the project's Chain of Custody as per MADEP's Interim Policy # COMM-94-007 and their subsequent Technical Update for freshwater sediment screening (May 2002).

1.2 Quality Assurance Planning Considerations

The end use of the data will determine the quality assurance requirements that are necessary to produce data of acceptable quality. These quality assurance requirements may be defined in a site-specific workplan or Quality Assurance Project Plan (QAPP) (hereafter referred to as the project plan) and may include duplicate or replicate measurements or confirmatory measurements.

2.0 RESPONSIBILITIES

Field personnel are responsible for verifying that all sampling equipment is in proper operating condition prior to use and for implementing the sampling procedures in accordance with this SOG and any specific project plan.

The project manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the measurements in accordance with this SOG and the project plan.

3.0 REQUIRED MATERIALS

The following materials may be necessary for this procedure:

- Sediment coring or grab sampling device
- Stainless steel mixing bowl
- Stainless steel mixing spoon or tool
- Nitrile gloves
- Alconox
- Pre-cleaned sample jars provided by laboratory
- Pencil and labeling marker or pen
- Field data sheets or logbooks
- GPS receiver and/or map of target waterbody to record sample locations

4.0 METHOD

Field personnel are to collect sediment cores or grabs in accordance with the instructions provided with each specific sampling device deployed. Nitrile gloves should be worn at all times during these procedures. At each sampling location, a pre-cleaned grab sample dredge or corer is to be deployed,

typically from a boat. All equipment is to be decontaminated using alconox and fresh water before the collection of each discrete sample. If specified by the project plan, samples may be composited in a pre-cleaned stainless steel mixing bowl and mixed thoroughly with a pre-cleaned stainless steel spoon before being transferred to the glass sampling jars provided by the laboratory. However, volatile organic compound (VOC) samples should be collected from cores prior to compositing.

The sample jar should be labeled with the sample identification, date, and any other project specific requirements. This information should be recorded in a field book at the time of sampling along with other essential information such as water depth, sample coordinates (or the location should be mapped on a figure at the time of sampling), and any other general notes on the nature of the sediment collected.

5.0 QUALITY CONTROL

Duplicate field samples or split samples may be collected if specified by the project plan. Once samples have been retrieved and placed into jars, the samples should be kept on ice or refrigerated until the laboratory can analyze them. Specific sample volumes, holding times, and detection limits for each parameter to be analyzed (Table 1) should be adhered to unless the project plan has outlined project-specific requirements.

TABLE 1. SEDIMENT ANALYSIS

Parameter	Volume Needed (ml)	Sample Container	Sample Preservation	Maximum Hold Time (hours)	Detection Limits (mg/Kg)	EPA #
Arsenic	100 g	Amber Glass	Ice	6 months	0.5	200.7
Cadmium	100 g	Amber Glass	Ice	6 months	0.1	200.7
Chromium	100 g	Amber Glass	Ice	6 months	1.0	200.7
Copper	100 g	Amber Glass	Ice	6 months	1.0	200.7
Lead	100 g	Amber Glass	Ice	6 months	1.0	200.7
Mercury	100 g	Amber Glass	Ice	6 months	0.02	245.1
Nickel	100 g	Amber Glass	Ice	6 months	1.0	200.7
Zinc	100 g	Amber Glass	Ice	6 months	1.0	200.7
PCBs	100 g	Amber Glass	Ice	7 days	0.01	8082
PAHs	100 g	Amber Glass	Ice	7 days	0.02	8270
EPH	100 g	Amber Glass	Ice	14 days	25	418.1
VOCs	100 g	Amber Glass	Methanol, Ice	7 days	0.1	EPA/ACE 8260
% Organic Content	100 g	Amber Glass	Ice	7 days	1.0%	160.4
% Ash Content	100g	Amber Glass	Ice	7 days	1.0%	160.4
Grain Size Analysis (Sieve and Hydrometer)	1,000g	Plastic Bag/Glass	None Required	Indefinite	0.1%	ASTMD 2216
% Water	100g	Amber Glass	Ice	14 days	1.0%	160.3

6.0 DOCUMENTATION

Documentation for recorded data must include a minimum of the following:

- Date and time of collection and analysis
- Signature or initials of person performing the collection or measurement
- Sample identification/station location
- Pertinent comments

7.0 TRAINING/QUALIFICATIONS

To properly perform sediment collections, the field personnel must be familiar with the techniques stated in this SOG and experienced in the operation of the sampling equipment.

8.0 REFERENCES

MADEP Interim Policy # COMM-94-007

MADEP 2002. Technical Update: Freshwater Sediment Screening Benchmarks for Use under the Massachusetts Contingency Plan. May 2002.

STANDARD OPERATING GUIDELINES FOR THE ACQUISITION OF SURFACE WATER

1.0 INTRODUCTION

1.1 Purpose and Applicability

This Standard Operating Guideline (SOG) provides basic instructions for the routine acquisition of surface water. The methods outlined below are intended (1) to standardize water sample collection methods used by ESS Group, Inc. (ESS) field personnel; (2) to ensure that samples delivered to the laboratory represent field conditions as accurately as possible; (3) to standardize recording of field data to assure proper documentation of sample collection; (4) to minimize cross contamination between sampling sites.

1.2 Quality Assurance Planning Considerations

The end use of the data will determine the quality assurance requirements that are necessary to produce data of acceptable quality. These quality assurance requirements will be defined in the site-specific workplan or Quality Assurance Project Plan (QAPP) (hereafter referred to as the project plan) or laboratory Quality Assurance Manual (QAM) and may include duplicate or replicate measurements or confirmatory analyses.

2.0 RESPONSIBILITIES

2.1 Project Manager

The project manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the measurements in accordance with this SOG and the project plan.

2.2 Field Personnel

The analyst is responsible for verifying that the sampling bottles are appropriately sanitized and contain the appropriate preservative for the desired laboratory analyses. Sample bottle caps should be securely in place to ensure that no contamination has occurred and that preservative has not been released.

3.0 REQUIRED MATERIALS

The following materials are necessary for the acquisition of surface water:

- Nitrile gloves
- Labeled sampling container provided from contracted laboratory, which is appropriately sanitized and contains the appropriate preservative for the desired analyses
- Laboratory or field data sheets or logbooks
- List of sites or locations of each site to be sampled

4.0 METHOD

4.1 Sample Handling, Preservation, and General Measurement Procedures

- Unless noted otherwise, surface water samples will be collected via direct grab methods.
- Upon entering a sampling location, ESS field personnel shall minimize disturbance to upstream waters and shall always sample water from the undisturbed upstream region. In addition, when wading in waterbodies, field personnel will try and disturb as little bottom sediment as possible.

- Sample collection shall precede the measurement of physical field parameters (such as turbidity, conductivity, dissolved oxygen, etc.) in order to minimize the risk of sediment disturbance and/or contamination.
- Clean rubber gloves shall be worn at each sampling location. Gloves shall be rinsed with distilled water prior to subsequent sample collection. When sampling multiple sites on the same date, gloves may be rinsed in the immediate downstream reaches of the waterbody to be sampled, before sample collection, in order to minimize the risk of cross-contamination. When warranted by the sensitivity of the laboratory analyses under investigation or at the Clients request, new, sterile rubber gloves shall be worn at each different sampling location.
- In absence of a project specific sampling protocol, grab samples are to be collected from beneath the water surface (at approximately 8 to 12 inches beneath the surface or mid-way between the surface and the bottom if the waterbody is shallow, (EPA 1997)). Samples will be collected at an appropriate distance from the stream bank or lake shoreline and away from submerged obstacles. For small streams (i.e., 10-20 feet wide with a maximum depth of less than 2 feet) the appropriate distance to collect a sample would be the center, while within larger streams the sample would be taken at a location where water depth is 2-3 feet.
- When collecting samples, ESS field personnel shall stand downstream of the desired sampling location, hold the bottle near its base and plunge it below the water surface with the opening (mouth) downward. The opening of sample bottles shall always be directed away from field personnel in an upstream direction.
- Sample containers with preservatives should not be used to collect surface water samples. If using containers with preservatives, a pre-cleaned container of similar type should be used to collect the sample with subsequent transfer to the preserved container.
- ESS personnel shall leave an approximate 1-inch air space (except for dissolved oxygen and BOD samples) in sample bottles, so that bottles may be shaken (if needed) before analyses (EPA, 1997).
- ESS personnel shall place sample bottles and temperature blanks (if required by QAPP or QAM) in a cooler filled with ice (if required by QAPP or QAM).
- The testing or analytical method and sample containers, preservation technique, and sample volumes should be selected in consultation with the laboratory to ensure that the samples obtained will provide the desired results.

5.0 QUALITY CONTROL

5.1 Field Duplicates

Field duplicate measurements of a single sample will be performed at the frequency specified in the project plan. Collection of duplicates will adhere to the surface water acquisition methods described above. Field duplicates will be collected immediately following initial sample collection.

6.0 DOCUMENTATION

Surface water quality field data will be reported in field notebooks by ESS personnel. Surface water quality laboratory data will be reported by contracted laboratories on official laboratory letterhead. Any unanticipated site-specific information, which requires ESS field personnel to deviate from the above SOG will be reported in an ESS field notebook. Documentation for recorded data must include a minimum of the following:

- Date and time of analysis
- Signature or initials of person performing the measurement
- Sample identification/station location
- Comments/observations

7.0 TRAINING/QUALIFICATIONS

To properly perform the acquisition of surface water, the analyst must be familiar with the sampling protocols as stated in this SOG.

8.0 REFERENCES

EPA, 1997. Volunteer Stream Monitoring: A Methods Manual. United States Environmental Protection Agency. Office of Water. EPA 841-B-97-003.

STANDARD OPERATING GUIDELINES FOR MEASUREMENT OF TEMPERATURE

1.0 INTRODUCTION

1.1 Purpose and Applicability

These Standard Operating Guidelines (SOG) provide basic instructions for routine measurement of temperature using any high quality mercury-filled thermometer or thermistor with analog or digital read-out device such as the Hydac Multimeter Probe and YSI Model 55. Multimeter instruments used for temperature measurement may measure additional parameters (e.g., dissolved oxygen, conductivity, pH, etc.). This SOG addresses temperature measurement only (other capabilities are outlined in the appropriate SOG). This SOG is designed specifically for the measurement of temperature in accordance with EPA Method 170.1 and Standard Method 2550 B which address thermometric temperature measurement of drinking, surface, and saline waters, and domestic and industrial wastes.

1.2 Quality Assurance Planning Considerations

The end use of the data will determine the quality assurance requirements that are necessary to produce data of acceptable quality. These quality assurance requirements will be defined in the site-specific workplan or Quality Assurance Project Plan (QAPP) (hereafter referred to as the project plan) or laboratory Quality Assurance Manual (QAM) and may include duplicate or replicate measurements or confirmatory measurements.

2.0 RESPONSIBILITIES

2.1 The analyst is responsible for verifying that the temperature measuring device is in proper operating condition prior to use and for implementing the calibration and measurement procedures in accordance with this SOG and the project plan.

2.2 The project manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the measurements in accordance with this SOG and the project plan.

3.0 REQUIRED MATERIALS

The following materials are necessary for this procedure:

- Thermometer or thermistor with analog or digital read-out device
- Manufacturer's instruction manual for the instrument
- National Institute of Standards and Technology (NIST)-traceable thermometer
- Laboratory or field data sheets or logbooks

4.0 METHOD

4.1 Sample Handling, Preservation, and General Measurement Procedures

To achieve accurate temperature measurements, samples should be analyzed immediately upon collection (preferably within 15 minutes). Samples should be collected in glass or plastic containers.

4.2 Calibration and Measurement Procedures

4.2.1 ESS-owned temperature measuring devices will, at a minimum, be checked annually as described in Section 5.0. The device will be checked against an NIST-traceable thermometer and the

necessary compensation made for the difference in temperature between the two. Rental equipment will be checked by the manufacturer and documentation provided to ESS.

4.2.2 Immerse the thermometer or temperature measuring device into the sample.

4.2.3 Swirl and take a reading when the value stabilizes.

4.2.4 Record the temperature reading to the nearest 0.50 for a thermometer or 0.10 for digital meter-type instruments. Compensate for any difference with the NIST-traceable thermometer.

4.2.5 Temperature data may be post-calibrated using any of a variety of calibration data including, but not limited to, field calibration points, manufacturer calibration data, and analytical results from samples collected during field deployment of the sensors. The decision criteria for post calibration, and the technique used, will be specified in the project plan, and will be consistent with the manufacturer's recommendations.

4.3 Troubleshooting Information

If there are any performance problems with any of the meter-type temperature measuring devices, consult the appropriate section of the meter instruction manual for the checkout and self-test procedures. If the problem persists, consult the manufacturer's customer service department immediately for further instructions. If a performance problem exists with the thermometer, discard the thermometer and replace it.

4.4 Maintenance

Instrument maintenance for meter-type temperature measuring devices should be performed according to the procedures and frequencies required by the manufacturer.

5.0 QUALITY CONTROL

5.1 The temperature measuring devices will, at a minimum, be checked against an NIST-traceable thermometer at the frequency stated in Section 4.2.1. This verification procedure will be performed as follows:

- Immerse the thermometer or temperature sensor and the NIST-traceable thermometer into a sample.
- Allow the readings to stabilize.
- Record the readings and document the difference.
- Label the thermometer or temperature sensor with the correction value/adjustment and the date the accuracy check was performed.
- Compensate for the difference when sample measurements are taken.

5.2 Duplicate measurements of a single sample will be performed at the frequency stated in the project plan. In the absence of project-specific criteria, duplicate measurements should agree within $\pm$ 0.50C or approximately $\pm$ 1.00F.

6.0 DOCUMENTATION

6.1 Records for checking the accuracy of the thermometer or temperature measuring device (where applicable) will include:

- Date
- Thermometer or meter-type temperature measuring device checked
- Reference thermometer number
- Readings for reference thermometer and thermometer being checked
- Adjustment made for difference in readings
- Initials of analyst

6.2 Documentation for recorded data must include a minimum of the following:

- Date and time of analysis
- Signature or initials of person performing the measurement
- Thermometer ID # or instrument identification number/model
- Sample identification/station location
- Temperature of sample (including units and duplicate measurements) compensated for any difference with the reference thermometer if applicable
- Comments

7.0 TRAINING/QUALIFICATIONS

To properly perform temperature measurements, the analyst must be familiar with the calibration and measurement techniques stated in this SOG. The analyst must also be experienced in the operation of the meter.

Certain state certification programs require that temperature measurements in the field be taken by, or in the presence of, personnel that are qualified under the certification program.

8.0 REFERENCES

Standard Methods for the Examination of Water and Wastewater, 17th Edition, 1989.

Methods for the Chemical Analysis of Water and Wastes, EPA 600/4-79-020, Revised 1983.

STANDARD OPERATING GUIDELINES FOR MEASUREMENT OF DISSOLVED OXYGEN

1.0 INTRODUCTION

1.1 Purpose and Applicability

These Standard Operating Guidelines (SOG) provide basic instructions for routine measurement of dissolved oxygen using a polarographic sensor equipped dissolved oxygen meter with a digital read-out such as the YSI Model 55 Handheld Dissolved Oxygen System. Measurements are made in accordance with EPA Standard Methods that addresses dissolved oxygen measurement of drinking, surface, and saline waters, and domestic and industrial wastes.

1.2 Quality Assurance Planning Considerations

The end use of the data will determine the quality assurance requirements that are necessary to produce data of acceptable quality. These quality assurance requirements will be defined in the site-specific workplan or Quality Assurance Project Plan (QAPP) (hereafter referred to as the project plan) or laboratory Quality Assurance Manual (QAM) and may include duplicate or replicate measurements or confirmatory measurements.

2.0 RESPONSIBILITIES

The analyst is responsible for verifying that the dissolved oxygen measuring device is in proper operating condition prior to use and for implementing the calibration and measurement procedures in accordance with this SOG and the project plan.

The project manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the measurements in accordance with this SOG and the project plan.

3.0 REQUIRED MATERIALS

The following materials are necessary for this procedure:

- Dissolved oxygen meter with digital read-out device
- Manufacturer's instruction manual for the instrument
- YSI Model 5775 Standard Membrane Kit with KCl solution and O-rings
- NIST-traceable thermometer

Laboratory or field data sheets or logbooks

4.0 METHOD

4.1 Sample Handling, Preservation, and General Measurement Procedures

To achieve accurate dissolved oxygen measurements, samples should be analyzed *in situ*. Measurements in flowing waters should be made in relatively turbulent free areas. Measurements in standing waters will require probe agitation to create water movement around the probe.

4.2 Calibration and Measurement Procedures

To accurately calibrate the YSI Model 55, you will need to know the approximate altitude of the region in which you are located and the approximate salinity of the water you will be analyzing. Fresh water has a salinity of approximately zero. Seawater has an approximate salinity of 35 parts per thousand (ppt). If uncertain, measure salinity with an appropriate device.

- Ensure that the sponge inside the instrument's calibration chamber is wet then insert the probe into the chamber. Turn the instrument on and wait for readings to stabilize (approximately 15 minutes).
- To calibrate, enter the calibration menu by pressing and releasing both the up and down arrow keys at the same time. Enter the altitude (in hundreds of feet) at the prompt by using the arrow keys to increase or decrease the altitude (example: 12 = 1,200 feet). Press enter when correct altitude is shown.
- The meter should display CAL in the lower left of the display with the calibration value in the lower right of the display and the current D.O. reading (before calibration) should be on the main display. Once the D.O. reading is stable, press ENTER. Enter the salinity at the prompt by using the arrow keys. Press ENTER when finished and the instrument will return to normal operation.
- Calibration should be performed at a temperature within ± 10°C of the sample temperature. Verify the calibration every 15 samples and at the end of the day.
- If erratic readings occur, replace membrane as per the manufacturer's manual. The average replacement interval is two to four weeks.
- Replace the membrane as per the manufacturer's manual if bubbles appear (>1/8 inch diameter), or if the membrane becomes damaged, wrinkled, or fouled.
- Avoid contact with any environment which contains substances that may attack the probe materials (e.g. acids, caustics, and strong solvents).
- The meter must be re-calibrated following any maintenance activities and prior to the next use.

4.3 Troubleshooting Information

If there are any performance problems with the dissolved oxygen-measuring device, consult the appropriate section of the instruction manual for the checkout and self-test procedures. If the problem persists, consult the manufacturer's customer service department immediately for further instructions.

4.4 Maintenance

Instrument maintenance for meter-type dissolved oxygen measuring devices should be performed according to the procedures and frequencies required by the manufacturer.

5.0 QUALITY CONTROL

Duplicate measurements of a single sample will be performed at the frequency specified in the project plan. In the absence of project-specific criteria, duplicate measurements should agree within ± 0.2 mg/L.

The temperature readout of the meter will be checked regularly (at least weekly) against a NIST-traceable thermometer. If the difference is greater than 0.5°C, the instrument manufacturer will be consulted for instructions. Temperature measurements will be compensated for any difference with the reference thermometer.

6.0 DOCUMENTATION

All dissolved oxygen meter calibration, checks, and maintenance information will be recorded on the daily calibration sheet or logbook. Dissolved oxygen data may be recorded on the appropriate laboratory or field data sheets or logbooks.

- Calibration documentation must be maintained in a thorough and consistent manner. At a minimum, the following information must be recorded:

- Date and time of calibration
- Signature or initials of person performing the measurement
- Instrument identification number/model
- Expiration dates and batch numbers for all standard solutions
- Readings for all continuing calibration checks
- Comments

- Documentation for recorded data must include a minimum of the following:
  - Date and time of analysis
  - Signature or initials of person performing the measurement
  - Instrument identification number/model
  - Sample identification/station location
  - Dissolved oxygen, both in mg/L and percent saturation (corrected for any difference with reference thermometer) and temperature of sample (including units and duplicate measurements)
  - Comments

7.0 TRAINING/QUALIFICATIONS

To properly perform dissolved oxygen measurements, the analyst must be familiar with the calibration and measurement techniques stated in this SOG. The analyst must also be experienced in the operation of the meter.

Certain state certification programs require that dissolved oxygen measurements in the field be taken by, or in the presence of, personnel that are qualified under the certification program.

8.0 REFERENCES

Standard Methods for the Examination of Water and Wastewater, 21st Edition, 2005.

Methods for the Chemical Analysis of Water and Wastes, EPA 600/4-79-020, Revised 1983.

STANDARD OPERATING GUIDELINES FOR MEASUREMENT OF PH

1.0 INTRODUCTION

1.1 Purpose and Applicability

These Standard Operating Guidelines (SOG) provide basic instructions for routine calibration and operation of a variety of pH meters, including the YSI Model 55, Hydac Multimeter Probe and the pHep pH Testers. Although these meters may measure additional parameters (e.g., temperature, specific conductivity, etc.), this SOG addresses pH measurement only (other capabilities are outlined in the appropriate SOG and manufacturer's individual instrument manuals). This SOG is designed specifically for the measurement of pH in accordance with EPA Method 150.1 and Standard Method 4500-H B which address electrometric pH measurements of drinking, surface, and saline waters, domestic and industrial wastes, and acid rain.

1.2 Quality Assurance Planning Considerations

The end use of the data will determine the quality assurance requirements that are necessary to produce data of acceptable quality. These quality assurance requirements will be defined in the site-specific workplan or Quality Assurance Project Plan (QAPP) (hereafter referred to as the project plan) or laboratory Quality Assurance Manual (QAM) and may include duplicate or replicate measurements or confirmatory analyses.

2.0 RESPONSIBILITIES

- The analyst is responsible for verifying that the pH meter is in proper operating condition prior to use and for implementing the calibration and measurement procedures in accordance with this SOG and the project plan.
- The project manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the measurements in accordance with this SOG and the project plan.

3.0 REQUIRED MATERIALS

The following materials may be necessary for this procedure:

- pH meter
- pH meter manufacturer's instruction manual
- Deionized water
- 4.0, 7.0, and 10.0 buffer solutions
- Lint-free tissues
- Mild detergent
- 10% hydrochloric acid
- National Institute of Standards and Technology (NIST)-traceable thermometer
- Calibration sheets or logbook
- Laboratory or field data sheets or logbooks

4.0 METHOD

4.1 Sample Handling, Preservation, and General Measurement Procedures

4.1.1 To achieve accurate pH measurements, samples should be analyzed in the field (preferably within 15 minutes), or as soon as possible after collection. Sample should be collected in plastic or glass containers.

4.1.2 After measuring a sample containing oily material or particulate matter, the electrode must be cleaned by carefully wiping with a lint-free cloth, or washing gently in a mild detergent, followed by a deionized water rinse. If this does not suffice, an additional rinse with 10% hydrochloric acid (followed by deionized water) may be needed.

4.1.3 As temperature can affect the pH measurements obtained, both the pH and the temperature of the sample must be recorded. Both the Hydac Multimeter and the pHep Tester that will be used in this study have the ability to compensate for temperature.

4.1.4 Calibration must include a minimum of two points that bracket the expected pH of the samples to be measured. Calibration measurements must be recorded in logbook.

4.1.5 Primary standard buffer salts available from NIST can be purchased and are necessary for situations where extreme accuracy is required. Secondary standard buffers may be purchased as a solution from commercial vendors and are recommended for routine use. Buffers should not be used after their expiration dates as provided by the manufacturer. An expiration date of one year should be used if the manufacturer does not supply an expiration date or if the buffers are prepared from pH powder pillows, etc.

4.1.6 When using the meter in the laboratory, always place the buffer/sample beaker on the magnetic stirrer, and make sure the stirring bar is rotating during measurements. Rinse the stirring bar as well as the beaker between buffers/samples.

EXCEPTION: Do not use the magnetic stirrer for acid rain samples. It is crucial not to induce dissolved gases into the sample to be absorbed or desorbed, as this will alter the pH. Stir the sample gently for a few seconds after introducing the electrode, then allow the electrode to equilibrate prior to recording temperature and pH readings.

4.1.7 When the meter is being used in the field, move the probe in a way that creates sufficient sample movement across the sensor; this insures homogeneity of the sample and suspension of solids. If sufficient movement has occurred, the readings will not drift (<0.l pH units). Rinse the electrode with deionized water between samples and wipe gently with a lint-free tissue.

4.1.8 When measuring the pH of hot liquids, wait for the liquid to cool to 160°F or below.

4.1.9 Fluctuating readings may indicate more frequent instrument calibrations are necessary.

4.2 Calibration and Measurement Procedures

4.2.1 The pH meter must be calibrated daily before any analyses are performed. The meter should be re-calibrated every 12 hours or at the frequency specified in the project plan.

4.2.2 Connect the electrode to the meter. Choose either 7.0 and 10.0 (high range) or 4.0 and 7.0 (low range) buffers, whichever will bracket the expected sample range. Place the buffer in a clean glass beaker. If the pH is being measured in a laboratory, place the beaker on the magnetic stirrer and place the stirring bar in the beaker. Measure and record the temperatures of the buffers using a calibrated thermometer or automatic temperature compensation (ATC).

4.2.3 Place the electrode into the 10.0 buffer or into the 7.0 buffer.

4.2.4 Adjust the instrument calibration according to the manufacturer's instructions. Discard the buffer and rinse the beaker and stirring bar thoroughly with deionized water.

4.2.5 Refill the beaker with the 7.0 buffer or the 4.0 buffer. Rinse the electrode, gently wipe with a lint-free tissue, and place it in the selected buffer solution. If the pH is being measured in a laboratory, place the beaker on the magnetic stirrer and place the stirring bar in the beaker. Continue adjusting the instrument calibration according to the manufacturer's instructions. Record the electrode slope (if provided by the instrument) on the calibration sheet (an acceptable slope is between 92 and 102 percent). Measure and record the temperature of the buffer using a calibrated thermometer or ATC. Discard the buffer and rinse the beaker and stirring bar thoroughly with deionized water.

4.2.6 An additional check may be performed, if required by the project plan, by placing the electrode into an additional buffer solution. This buffer should be from a different source than the buffers used for the initial calibration. This buffer should read within +0.2 pH units of the buffer's true pH value.

4.2.7 Verify the calibration every 15 samples and at the end of the day. Recalibrate the instrument if the check value varies more than 0.2 pH units from the true value.

4.2.8 The electrode will be rinsed with deionized water and wiped gently with a lint-free tissue between sample analysis.

4.2.9 Recalibrate the instrument if the buffers do not bracket the pH of the samples.

4.2.10 The meter must be re-calibrated following any maintenance activities and prior to the next use.

4.3 Troubleshooting Information

If there are any performance problems with any of the pH meters which result in the inability to achieve the acceptance criteria presented in Section 5.0, consult the appropriate section of the meter instruction manual for the checkout and self-test procedures. If the problem persists, consult the manufacturer's customer service department immediately for further instructions.

4.4 Maintenance

4.4.1 Instrument maintenance should be performed according to the procedures and frequencies required by the manufacturer.

4.4.2 The electrode must be stored and maintained according to the manufacturer's instructions.

4.4.3 If an instrument with ATC is being used, the device should be checked on a quarterly basis for accuracy with an NIST thermometer.

5.0 QUALITY CONTROL

5.1 Duplicate measurements of a single sample will be performed at the frequency specified in the project plan. In the absence of project-specific criteria, duplicate measurements should agree within ±0.l pH units.

5.2 The temperature readout of the meter will be checked annually against an NIST-traceable thermometer. If the difference is greater than 0.2°C, the instrument manufacturer will be consulted for instructions. Temperature measurements will be compensated for any difference with the reference thermometer.

5.3 Some regulatory agencies may require the analysis of USEPA Water Supply (WS) or Water Pollution (WP) performance evaluation samples. These performance evaluation samples will be analyzed as required.

6.0 DOCUMENTATION

6.1 All pH meter calibration, temperature check, and maintenance information will be recorded on the daily calibration sheet (Figure 1). pH data may be recorded on the appropriate laboratory or field data sheets or logbooks.

6.2 Calibration documentation must be maintained in a thorough and consistent manner. At a minimum, the following information must be recorded:

- Date and time of calibration
- Signature or initials of person performing the measurement
- Instrument identification number/model
- Expiration dates and batch numbers for all buffer solutions
- Reading for pH 7.0 buffer before and after meter adjustment
- Reading for pH 4.0 or 10.0 buffer before and after meter adjustment
- Readings for all continuing calibration checks
- Temperature of buffers (corrected for any difference with reference thermometer), including units
- Comments

6.3 Documentation for recorded data must include a minimum of the following:

- Date and time of analysis
- Signature or initials of person performing the measurement
- Instrument identification number/model
- Sample identification/station location
- Temperature (corrected for any difference with reference thermometer) and pH of sample (including units and duplicate measurements)
- Comments

7.0 TRAINING/QUALIFICATIONS

To properly perform pH measurements, the analyst must be familiar with the calibration and measurement techniques stated in this SOG. The analyst must also be experienced in the operation of the meter.

Certain state certification programs require that pH measurements in the field be taken by, or in the presence of, personnel that are qualified under the certification program.

8.0 REFERENCES

Standard Methods for the Examination of Water and Wastewater, 17th Edition, 1989.

Methods for the Chemical Analysis of Water and Wastes, EPA 600/4-79-020, Revised 1983.

STANDARD OPERATING GUIDELINES FOR MEASUREMENT OF SPECIFIC CONDUCTANCE

1.0 INTRODUCTION

1.1 Purpose and Applicability

These Standard Operating Guidelines (SOG) provide basic instructions for routine calibration and operation of a variety of specific conductance meters. Although this meter measures additional parameters (e.g., temperature, TDS), this SOG addresses specific conductance measurement only (other capabilities are outlined in the appropriate SOG and manufacturer's individual instrument manuals). This SOG is designed specifically for the measurement of specific conductance in accordance with EPA Method 120.1 and Standard Method 2510 B which address specific conductance measurements of drinking, surface, and saline waters, domestic and industrial wastes, and acid rain.

1.2 Quality Assurance Planning Considerations

The end use of the data will determine the quality assurance requirements that are necessary to produce data of acceptable quality. These quality assurance requirements will be defined in the site-specific workplan or Quality Assurance Project Plan (QAPP) (hereafter referred to as the project plan) or laboratory Quality Assurance Manual (OAM) and may include duplicate or replicate measurements or confirmatory analyses.

2.0 RESPONSIBILITIES

The analyst is responsible for verifying that the specific conductance meter is in proper operating condition prior to use and for implementing the calibration and measurement procedures in accordance with this SOG and the project plan.

The project manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the measurements in accordance with this SOG and the project plan.

3.0 REQUIRED MATERIALS

The following materials are necessary for this procedure:

- Specific conductance meter
- Specific conductance meter manufacturer's instruction manual
- Deionized water
- KCl standard at concentration that approximates sample concentrations
- Lint-free tissues
- National Institute of Standards and Technology (NIST)-traceable thermometer
- Calibration sheets or logbook
- Laboratory or field data sheets or logbooks

4.0 METHOD

4.1 Sample Handling, Preservation, and General Measurement Procedures

- Specific conductance measurements should be taken soon after sample collection since temperature changes, precipitation reactions, and absorption of carbon from the air can affect the specific

conductance. If specific conductance measurements cannot be taken immediately (within 24 hours), samples should be filtered through a 0.45 μm filter, stored at 4°C and analyzed within 28 days.

- Report results as specific conductance, μmhos/cm at 25°C.
- As temperature can affect the specific conductance measurements obtained, record both the specific conductance and the temperature of the sample. The Cole-Parmer Portable Conductivity Meter and YSI Model 85 have the ability to compensate for temperature.
- Secondary standards may be purchased as a solution from commercial vendors. These standards should not be used after their expiration dates as provided by the manufacturer. An expiration date of one year should be used if the manufacturer does not supply an expiration date or if the standards are prepared from various salts (e.g., KCl).

4.2 Calibration and Measurement Procedures

- The specific conductance meter must be calibrated daily (or the calibration checked) before any analyses are performed.
- Set up the instrument according to the manufacturer's instructions.
- Rinse the probe with deionized water and dry with a lint-free tissue.
- Dip the probe into the calibration standard. Immerse the probe tip beyond the upper steel band. Stir the probe gently to create a homogenous sample.
- Record the stabilized specific conductance reading of the standard and the temperature. Enter the calibration mode (according to manufacturer's instructions) and change the value on the primary display to match the value of the calibration standard. The meter can be adjusted to ± 20% from the default setting. If the measurement differs by more than ± 20%, the probe should be cleaned or replaced as needed. If the meter does not have automatic temperature compensation (ATC), correct all measurements to 25°C by adding 2% of the reading per degree if the temperature is below 25°C or by subtracting 2% of the reading per degree if the temperature is above 25°C.
- An additional check may be performed, if required by the project plan, by placing the probe into an additional KCl standard. This standard should be from a different source than the standard used for the initial calibration. This standard should read within 5% of the true value.
- Verify the calibration every 15 samples and at the end of the day. Recalibrate or replace the instrument if the check value is not within 15% of the true value.
- The probe will be rinsed with deionized water and wiped gently with a lint-free tissue between sample analyses.
- The meter must be recalibrated following any maintenance activities and prior to the next use.
- Conductivity data may be post calibrated using any of a variety of calibration data including, but not limited to field calibration points, manufacturer calibration data, and analytical results from samples collected during field deployment of the sensors. The decision criteria for post calibration, and the technique used will be specified in the project plan, and will be consistent with the manufacturer's recommendations.

4.3 Troubleshooting Information

If there are any performance problems with any of the specific conductance meters which result in inability to achieve the acceptance criteria presented in Section 5.0, consult the appropriate section of the

meter instruction manual for the checkout and self-test procedures. If the problem persists, consult the manufacturer's customer service department immediately for further instructions.

4.4 Maintenance

- Instrument maintenance should be performed according to the procedures and frequencies required by the manufacturer.
- The probe must be stored and maintained according to the manufacturer's instructions.
- If an instrument with ATC is being used, the meter should be checked annually for accuracy with an NIST thermometer.

5.0 QUALITY CONTROL

- The meter must be calibrated daily before sampling and recalibrated every 12 hours, and will not be used for sample determinations of specific conductance unless the initial check standard value is within 5% of the true value.
- Duplicate measurements of a single sample will be performed at the frequency specified in the project plan. In the absence of project-specific criteria, duplicate measurements should agree within 10%.
- The temperature readout of the meter will be checked against an NIST traceable thermometer at least quarterly. If the difference is greater than 0.2°C, the instrument manufacturer will be consulted for instructions. Temperature measurements will be compensated for any difference with the reference thermometer.
- Some agencies may require the analysis of USEPA Water Pollution (WP) performance evaluation samples. These performance evaluation samples will be analyzed as required.

6.0 DOCUMENTATION

- All specific conductance meter calibration, temperature check, and maintenance information will be recorded on the daily calibration sheet (an example is presented as Figure 1). Specific conductivity data may be recorded on the appropriate laboratory or field data sheets or logbooks.
- Calibration documentation must be maintained in a thorough and consistent manner. At a minimum, the following information must be recorded:
    - Date and time of calibration
    - Signature or initials of person performing the measurement
    - Instrument identification number/model
    - Expiration dates and batch numbers for all standards
    - Reading for standard before and after meter adjustment
    - Readings for all continuing calibration checks
    - Temperature of standards (corrected for any difference with reference thermometer)
    - Comments
- Documentation for recorded data must include a minimum of the following:
    - Date and time of analysis
    - Signature or initials of person performing the measurement

- Instrument identification number/model
- Sample identification/station location
- Temperature (corrected for any difference with reference thermometer) and conductance of sample (including units and duplicate measurements) Note: show all calculations for converting instrument reading to μmhos/cm if the instrument provides readings in any other units. Useful conversions are: 1 mS/m = 10 μmho/cm or 1 μmho/cm = 0.1 mS/m.
- Comments

7.0 TRAINING/QUALIFICATIONS

To properly perform specific conductance measurements, the analyst must be familiar with the calibration and measurement techniques stated in this SOG. The analyst must also be experienced in the operation of the meter.

Certain state certification programs require that specific conductance measurements be taken in the field by, or in the presence of, personnel that are qualified under the certification program.

8.0 REFERENCES

Standard Methods for the Examination of Water and Wastewater, 17th Edition, 1989.

Methods for the Chemical Analysis of Water and Wastes, EPA 600/4-79-020, Revised 1983.

STANDARD OPERATING GUIDELINESFOR MEASUREMENT OF TURBIDITY

1.0 INTRODUCTION

1.1 Purpose and Applicability

These Standard Operating Guidelines (SOG) provide basic instructions for routine measurement of turbidity using a nephelometric turbidity meter with a digital read-out device such as the LaMotte 2020 Turbidimeter. Measurements are made in accordance with EPA Method 180.1 that addresses nephelometeric turbidity measurement of drinking, surface, and saline waters, and domestic and industrial wastes.

1.2 Quality Assurance Planning Considerations

The end use of the data will determine the quality assurance requirements that are necessary to produce data of acceptable quality. These quality assurance requirements will be defined in the site-specific workplan or Quality Assurance Project Plan (QAPP) (hereafter referred to as the project plan) or laboratory Quality Assurance Manual (QAM) and may include duplicate or replicate measurements or confirmatory measurements.

2.0 RESPONSIBILITIES

2.1 The analyst is responsible for verifying that the turbidity measuring device is in proper operating condition prior to use and for implementing the calibration and measurement procedures in accordance with this SOG and the project plan.

2.2 The project manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the measurements in accordance with this SOG and the project plan.

3.0 REQUIRED MATERIALS

The following materials are necessary for this procedure:

- Turbidity meter with digital read-out device
- Manufacturer's instruction manual for the instrument
- Turbidity tubes
- Mild detergent
- Lint-free cloth
- Distilled water
- Nephelometric Turbidity Unit (NTU) calibration standards (1.00 NTU and 10.0 NTU)
- Laboratory or field data sheets or logbooks

4.0 METHOD

4.1 Sample Handling, Preservation, and General Measurement Procedures

To achieve accurate turbidity measurements, samples should be analyzed immediately upon collection (preferably within 15 minutes). Samples should be collected in glass or plastic containers.

4.2 Calibration and Measurement Procedures

4.2.1 Select a turbidity standard in the range of the samples to be tested (1.00 NTU or 10.0 NTU). Fill a turbidity tube with the standard, cap, and wipe the tube with the clean lint-free cloth.

4.2.2 Place the sample into the turbidity meter such that the indexing arrow on the turbidity tube is aligned with the indexing arrow on the meter face. Close the lid and press the "READ" button. If the displayed value is not the same as the value of the standard (within 2%), continue with the calibration procedure.

4.2.3 Follow the calibration procedures outlined by the manufacturer's manual.

4.2.4 Verify the calibration every 15 samples and at the end of the day. Recalibrate the instrument if the check value varies more than 2% from the true value.

4.2.5 The turbidity tubes will be rinsed with deionized water and wiped gently with a lint-free tissue between sample analysis.

4.2.6 Recalibrate the instrument with the appropriate NTU standard if the standard is not of the same order of magnitude as the samples being tested.

4.2.7 The meter must be re-calibrated following any maintenance activities and prior to the next use.

4.2.8 Record the turbidity reading to the nearest 0.01 NTU for measurements less than 11 NTU and to the nearest 0.1 for measurements greater than 11 NTU but less than 110 NTU. For values greater than 110 NTU record to the nearest 1 NTU.

4.3 Troubleshooting Information

If there are any performance problems with any of the meter-type turbidity measuring devices, consult the appropriate section of the meter instruction manual for the checkout and self-test procedures. If the problem persists, consult the manufacturer's customer service department immediately for further instructions.

4.4 Maintenance

Instrument maintenance for meter-type turbidity measuring devices should be performed according to the procedures and frequencies required by the manufacturer.

5.0 QUALITY CONTROL

5.1 The turbidity measuring tubes will, at a minimum, be checked against NTU calibration standards at the frequency stated in Section 4.2.1. This verification procedure will be performed as follows:

- Insert the turbidity tube with distilled water into the turbidity meter.
- Press "READ".
- Record the readings and document the difference.
- Label each turbidity tube with its corresponding turbidity correction value.
- Record the adjustment and the date the accuracy check was performed in a logbook.
- Compensate for the difference when sample measurements are taken.

5.2 Duplicate measurements of a single sample will be performed at the frequency stated in the project plan. In the absence of project-specific criteria, duplicate measurements should agree within ± 2% for readings below 100 NTU and ± 3% for readings above 100 NTU.

6.0 DOCUMENTATION

All turbidity meter calibration, checks, and maintenance information will be recorded on the daily calibration sheet or logbook. Turbidity data may be recorded on the appropriate laboratory or field data sheets or logbooks.

6.1 Calibration documentation must be maintained in a thorough and consistent manner. At a minimum, the following information must be recorded:

- Date and time of calibration
- Signature or initials of person performing the measurement
- Instrument identification number/model
- Expiration dates and batch numbers for all standard solutions
- Reading for 1.00 NTU standard before and after meter adjustment
- Reading for 10.0 NTU standard before and after meter adjustment
- Readings for all continuing calibration checks
- Comments

6.2 Documentation for recorded data must include a minimum of the following:

- Date and time of analysis
- Signature or initials of person performing the measurement
- Instrument identification number/model
- Sample identification/station location
- Turbidity of sample (including units and duplicate measurements)
- Comments

7.0 TRAINING/QUALIFICATIONS

To properly perform turbidity measurements, the analyst must be familiar with the calibration and measurement techniques stated in this SOG. The analyst must also be experienced in the operation of the meter.

Certain state certification programs require that turbidity measurements in the field be taken by, or in the presence of, personnel that are qualified under the certification program.

8.0 REFERENCES

Standard Methods for the Examination of Water and Wastewater, 17th Edition, 1989.

Methods for the Chemical Analysis of Water and Wastes, EPA 600/4-79-020, Revised 1983.

STANDARD OPERATING GUIDELINES FOR MEASUREMENT OF WATER CLARITY WITH A SECCHI DISC

1.0 INTRODUCTION

This Standard Operating Guideline (SOG) provides basic instructions for the routine measurement of water clarity in lakes and ponds with a Secchi disc. Water clarity is a function of the number of particles in the water (algae, sediment, etc) and the color of the water, which both have an impact on the depth of light penetration. The transparency of the water column can be used as an indicator of water body productivity, with certain exceptions (e.g., naturally sediment laden waterbodies). Generally, the more productive a system is the more algae in the water column, and the lower the transparency. Water transparency can also be affected by erosionally suspended particles which are related to water depth and wave action. Thus on any given day the turbidity of a water body may be affected by its productivity, the season, wind speed and level of sunlight. The methods outlined below are intended (1) to standardize the use of a Secchi disc in the measurement of turbidity; (2) to standardize recording of field data to assure proper documentation of weekly, monthly and seasonal patterns in turbidity.

2.0 REQUIRED MATERIALS

The following materials are necessary for the measurement of turbidity with a Secchi disc:

- Weighted Secchi disc with attached length of rope marked off in one tenth of a meter increments with indelible ink.
- Field data sheets

3.0 METHODS

- A location will be selected from which to measure turbidity. This location will stay constant throughout the study.
- The date, weather conditions, and personnel conducting the measurement will be recorded on the field sheet.
- The Secchi disc will be lowered slowly into the water by the rope so that the weight enters the water first and the disc follows, flat side parallel to the water surface.
- The disc will continue to be lowered through the water column until it is no longer visible.
- A note will be made of the depth of the disc at this point in tenths of a meter by reading where the surface of the water touches the rope.
- The disc will then be slowly raised until it is just visible again.
- Once again a note will be made of the depth of the disc at this point.
- An average of these two depths will be calculated to give the “Secchi depth”, i.e. a measure of the turbidity of the water.

4.0 DOCUMENTATION

Secchi depth data will be reported on field data sheets for every day that a measurement is taken. Documentation for recorded data must include a minimum of the following:

- The date
- The time
- Weather Conditions
- Signature or initials of person performing the measurement
- Depth measurements and average Secchi depth
- Field comments/observations on anything that may influence the Secchi depth measurement that day.

STANDARD OPERATING GUIDELINES FOR THE CREATION OF AN AQUATIC PLANT MAP

1.0 INTRODUCTION

1.1 Purpose and Applicability

This Standard Operating Guideline (SOG) provides basic instructions for the mapping of aquatic plants present within standing waterbodies. The methods outlined below are intended to, (1) standardize plant mapping techniques used by ESS Group, Inc. (ESS) field personnel; and (2) standardize recording of field data to assure the creation of an accurate plant map.

2.0 RESPONSIBILITIES

2.1 Project Manager

The project manager is responsible for ensuring that project-specific requirements are communicated to the project team and for providing the materials, resources, and guidance necessary to perform the survey in accordance with this SOG and the project plan.

2.2 Field Personnel

The surveyors are responsible for identifying dominant aquatic plant beds within the waterbody, establishing the locations of the beds using GPS, noting the percentage of plant cover and biovolume throughout the waterbody, keeping a species list of all plants identified within the waterbody and collecting clearly marked samples of all those plants unidentifiable in the field.

3.0 REQUIRED MATERIALS

The following materials are necessary (unless otherwise noted) for the creation of a plant map:

- Boat
- Long handled grappling rake
- Throw grappling rake (for deeper waters)
- Aquascope
- Plant keys
- Enlarged outline of the waterbody on water resistant paper
- Water resistant field notebook
- Small see-through plastic bags
- Indelible marker
- Cooler
- Ice
- GPS unit (Trimble GeoExplorer 2005 series recommended)
- Underwater camera (Optional – useful in deeper waters)

4.0 METHOD

4.1 Aquatic Plant Survey and Sample Collection

A number of transects will be drawn on the map of the waterbody to act as a guide for the survey. The number and location of transects selected will depend on the size and shape of the waterbody, with the aim of thoroughly characterizing the plants within it.

The boat will be driven along each transect; at pre-determined points along each transect, anchor will be dropped and a detailed survey of the aquatic plants will be carried out in the immediate area. The number of points surveyed along each transect will depend on the bathymetry and plant diversity in the survey area, with the aim of characterizing changes in the composition, cover and biovolume of plant beds. Each point sampled along each transect will be numbered and recorded on the site map in order to link plant survey data with location information. Alternatively, records may be added electronically in the field, if this function is supported by the GPS unit used.

At each survey point a grappling rake will be used to sample aquatic plants from within the water column and the floor of the waterbody for closer identification.

Each plant present within each sample will be identified *in situ* (using keys if necessary) and recorded in the species list for the waterbody. The dominant plant at each transect point will be noted with its associated transect and point number in the field notebook.

If identification of certain plants is not possible in the field, a generous sample of these plants will be stored with a little water in a plastic bag clearly labeled with the associated transect and point number in indelible ink. All such sample bags will be stored in a cooler filled with ice to preserve the quality of the samples, and transported back to the lab for identification using a dissecting microscope, if necessary. Unknown plants will be assigned a code number (e.g. UK1) to use as species identification for future transects and sampling locations.

4.2 Assessment of Percentage Plant Cover and Percentage Plant Biomass

At each survey point ESS field personnel will use general observation as well as an Aquascope to estimate the percentage plant cover (i.e. the percentage of the bottom covered by plants, which is a factor of plant density). A simple code system will be used whereby percentage “ranges” are assigned an integer: i.e. 0 = 0%; 1 = 1%-25%; 2 = 26%-50%; 3 = 51%-75%; 4 = 76%-100%. At each survey point the estimation of plant cover will be recorded with the associated transect and point number in the field notebook. All estimations of plant cover and biomass are made by the same field personnel to ensure consistency.

In addition to plant cover, biovolume will be estimated by ESS field personnel at each survey point, using both general observation as well as an Aquascope (or underwater camera for deeper water). The percentage of biovolume represents that percentage of the water column that is occupied by plants; biovolume is a factor of water depth, plant height, and plant density. As noted above, a simple code system will be used to assign integers as estimations of percent biovolume. At each survey point the estimation of biovolume will be recorded with the associated transect and point number in the field notebook. All estimations of plant cover and biomass are made by the same field personnel to ensure consistency.

Assessment of both plant cover and biovolume will be made along the length of each transect with general observation and an Aquascope. In increased water depths or under turbid conditions, the grappling rake will be used to assess these measurements. The bottom of the waterbody will be scraped in order to estimate plant cover and biovolume. At depths greater than 16ft, the grappling rake will not be effective and the plant cover and biovolume will be assumed to be 0%.

4.3 Creation of Plant Maps

Upon completion of the field survey, dominant plant beds identified within the waterbody will be linked with associated transects and survey point locations to create a dominant aquatic plant distribution map.

Percentage plant cover and plant biovolume "code numbers" will be linked with the transects and survey point locations drawn onto the outline map to create maps that illustrate the percentage cover and percentage biomass of aquatic plants in every part of the waterbody.

5.0 QUALITY CONTROL

Dominant species as well as unidentifiable plants (unknowns) will be sampled *in situ* and transported back to the lab in plastic bags. Identification checks with other plant keys and consultations with ESS plant experts will be made to confirm species identification.

6.0 DOCUMENTATION

All observed and sampled plants will be recorded by ESS personnel in field notebooks in the form of a species list. Dominant plants will be also be associated with location information in the form of transect numbers and survey points. Transect lines and survey points will be recorded on a map outline of the waterbody that has been printed on water resistant paper (e.g. Rite-in-the-Rain). Any unanticipated site-specific information, which requires ESS field personnel to deviate from the above SOG will be reported in an ESS field notebook. Documentation for recorded data must include a minimum of the following:

- Survey date
- Weather conditions
- Signature or initials of person performing the survey
- Plant survey transect and point locations
- Comments/observations

Additionally, survey point data may be added electronically in the field using a GPS unit.

7.0 TRAINING/QUALIFICATIONS

To properly complete an assessment of plants within a waterbody, the analyst must be familiar with the sampling protocols as stated in this SOG, must have confidence in the use of plant keys and must have familiarity with the aquatic plants of the area in question.

Appendix C

Laboratory Quality Assurance Plans and Standard Operating Procedures

QUALITY MANUAL

Revision 2.11

Quality Manual

Revision 2.11
Effective: April 20, 2012

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Implemented by: R Warila
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General Manager

This manual contains laboratory policies and operational procedures as applied in all departments of Premier Laboratory, Inc.

Distribution Control Page

This manual is a controlled copy only if it bears a controlled copy number and original signature of the Premier Laboratory, Inc., Quality Assurance Officer. The Quality Assurance Officer is responsible for maintaining, updating, and distributing this manual throughout the laboratory.

Controlled Copy Number: _____

Issued to (name and affiliation): _____

Signature of Quality Assurance Officer: _____

Expiration date: _____

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TABLE OF CONTENTS

Scope of the Quality Systems Manual

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

This Quality Systems Manual contains quality assurance information necessary to implement the Premier Laboratory, Inc., Quality Assurance Program. All Premier Laboratory, Inc. management and analytical personnel are required to read and sign this manual, and are responsible for implementation of the Quality Assurance Program within their respective disciplines.

This manual does not however, include all the information necessary for complete implementation of the Quality Assurance Program. Additional information and procedures are found in the following:

1. *Statement of Qualifications:* Information about Premier Laboratory, Inc. facilities, instrumentation, organization, and resumes for key personnel.
2. *Client Services Manual:* Procedures for sample management, including: sample log-in, storage, and chain of custody.
3. *Standard Operating Procedures for Analyses:* Method-specific quality control requirements.
4. *Premier Laboratory, Inc. LIMS Manual:* Developed internally for use with Premier Laboratory, Inc.'s LIMS system.
5. *Hazardous Waste Management Document*: Developed in conjunction with various consultants for Premier Laboratory, Inc.'s waste disposal program.
6. *Chemical Hygiene Plan*: Developed in conjunction with a certified industrial hygienist for Premier Laboratory, Inc..
7. *EPA-815-R-05-004, Manual for the Certification of Laboratories Analyzing Drinking Water, January 2005.*
8. *Quality Systems, National Environmental Laboratory Accreditation Conference, June 2003.*

It is the responsibility of the Quality Assurance Officer to produce, maintain, update, and distribute the Quality Systems Manual at Premier Laboratory, Inc.. The Quality Systems Manual is updated yearly. The Quality Assurance Officer, in conjunction with the appropriate managers, will review the manual and make recommendations for changes. All analysts and managers are required to read, and acknowledge receipt of the Quality Systems Manual by signing the QM acknowledgement logbook maintained in the QA office.

Glossary of Definitions

Prepared by: _____ Reviewed and Implemented by: _____
Laboratory Director General Manager

1. **Accreditation**: The process by which an agency or organization evaluates and recognizes a program of study or an institution as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.
2. **Accrediting Authority**: The agency having responsibility and accountability for environmental laboratory accreditation and who grants accreditation.
3. **Accuracy**: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.
4. **Analytical Detection Limit**: The smallest amount of an analyte that can be distinguished in a sample by a given measurement procedure throughout a given confidence interval.
5. **Analytical Reagent (AR) Grade**: Designation for the high purity of certain chemical reagents and solvents given by the American Chemical Society.
6. **Assessor Body**: The organization that actually executes the accreditation process, i.e., receives and reviews accreditation applications, reviews QA documents, reviews proficiency testing results, surveys the site, etc., whether EPA, the state, or contracted private party.
7. **Batch**: Environmental samples, which are prepared and / or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same NELAC-defined matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group using the same calibration curve or factor. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.
8. **Blank**: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.
9. **Blind Sample**: A subsample for analysis with a composition known to the submitter. The analyst / laboratory may know the identity of the sample but not its composition. It is used to test the analyst or laboratory's proficiency in the execution of the measurement process.

10. **Calibrate**: To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other device, or the correct value for each setting of a control knob. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements.
11. **Calibration**: The set of operations which establish, under specified conditions, the relationship between values indicated by a measuring instrument or measuring system, or values represented by a material measure, and the corresponding known values of a measurement.
12. **Calibration Curve**: The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their analytical response.
13. **Calibration Standard**: A substance or reference material used to calibrate an instrument.
14. **Certified Reference Material (CRM)**: A reference material, one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body.
15. **Chain of Custody**: An unbroken trail of accountability that documents the physical security of samples, data and records.
16. **Confirmation**: Verification of the presence of a component through the use of an approach different from the original test method. These may include:
    - Second column confirmation
    - Alternate wavelength
    - Derivatization
    - Mass spectral interpretation
    - Alternative detectors or
    - Additional cleanup procedures.
17. **Corrective Action**: Action taken to eliminate the causes of an existing nonconformity, defect, or other undesirable situation in order to prevent recurrence.
18. **Data Audit**: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e., that they meet specified acceptance criteria.)
19. **Data Reduction**: The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useful form.
20. **Detection Limit**: The lowest concentration or amount of the target analyte that can be determined to be different from zero by a single measurement at a stated degree of confidence.
21. **Document Control**: The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly

and controlled to ensure use of the correct version at the location where the prescribed activity is performed.

22. **Duplicate Analyses**: The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.
23. **Environmental Detection Limit (EDL)**: The smallest level at which a radionuclide in an environmental medium can be unambiguously distinguished for a given confidence interval using a particular combination of sampling and measurement procedures, sample size, analytical detection limit, and processing procedure. The EDL shall be specified for the 0.95 or greater confidence interval. The EDL shall be established initially and verified annually for each test method and sample matrix.
24. **Holding Times (Maximum Allowable Holding Times)**: The maximum times that samples may be held prior to analysis and still be considered valid.
25. **Initial Demonstration of Capability**: Procedure to establish the ability of the laboratory to generate acceptable accuracy and precision.
26. **Internal Standard**: A known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the precision and bias of the applied analytical test method.
27. **Laboratory**: A body that calibrates and/or tests.

    Note:

    1. In cases where a laboratory forms part of an organization that carries out other activities besides calibration and testing, the term "laboratory" refers only to those parts of that organization that are involved in the calibration and testing process.
    2. As used herein, the term "laboratory" refers to a body that carries out calibration or testing
        - At or from a permanent location,
        - At or from a temporary facility, or
        - In or from a mobile facility.
28. **Laboratory Control Sample (however named, such as laboratory fortified blank or spiked blank)**: A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.
29. **Laboratory Duplicate**: Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.

30. **Legal Chain of Custody (COC)**: An unbroken trail of accountability that ensures the physical security of samples, data and records.
31. **Limit of Detection (LOD)**: The lowest concentration level that can be determined by a single analysis and with a defined level of confidence to be statistically different from a blank.
32. **Manager (however named)**: The individual designated as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual.
33. **Matrix**: The component or substrate, which contains the analyte of interest. For purposes of batch determination, the following matrix types shall be used:
    - **Aqueous**: Any aqueous sample excluded from the definition of a drinking water matrix or saline/estuarine source. This includes surface water, groundwater and effluents.
    - **Drinking water**: Any aqueous sample that has been designated a potable or potential potable water source.
    - **Saline / Estuarine**: Any aqueous sample from an ocean or estuary, or other salt-water source such as the Great Salt Lake.
    - **Non-aqueous liquid**: Any organic liquid with >15% settleable solids.
    - **Biological Tissue**: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.
    - **Solids**: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.
    - **Chemical Waste**: A product or by-product of an industrial process that results in a matrix not previously defined.
    - **Air Samples**: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter or other device.
34. **Matrix Spike (spiked sample, fortified sample)**: Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.
35. **Matrix Spike Duplicate (spiked sample, fortified sample duplicate)**: A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.
36. **May**: Permitted, but not required.
37. **Method Blank**: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples containing an analyte of interest through all steps of the analytical procedures.

38. **Method Detection Limit**: The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.
39. **Must**: Denotes a requirement that must be met.
40. **Negative Control**: Measures taken to ensure that a test, it's components, or the environment do not cause undesired effects, or produce incorrect test results.
41. **NELAC**: National Environmental Laboratory Accreditation Conference. A voluntary organization of state and federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories.
42. **NELAP**: The overall National Environmental Laboratory Accreditation Program of which NELAC is a part.
43. **Performance Audit**: The routine comparison of independently obtained quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.
44. **Performance Based Measurement System (PBMS)**: A set of processes wherein the data quality needs, mandates, or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner.
45. **Positive Control**: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.
46. **Precision**: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.
47. **Preservation**: Refrigeration and or reagents added at the time of sample collection to maintain the chemical and or biological integrity of the sample.
48. **Proficiency Test Sample (PT)**: A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst / laboratory can produce analytical results within specified acceptance criteria.
49. **Proficiency Testing**: Determination of the laboratory calibration or testing performance by means of inter-laboratory comparisons.
50. **Proficiency Testing Program**: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results in comparison to peer laboratories and the collective demographics and results summary of all participating laboratories.
51. **Protocol**: A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) which must be strictly followed.

52. **Pure Reagent Water**: Shall be water in which no target analytes or interferences are present at a concentration which would impact the results when using a particular analytical test method.
53. **Quality Assurance**: An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.
54. **Quality Control**: The overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.
55. **Quality Manual**: A document stating the quality policy, quality system, and quality practices of an organization. This may be also called a Quality Assurance Plan or a Quality Plan. The quality manual may call up other documentation relating to the laboratory's quality arrangements.
56. **Quality System**: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC.
57. **Quantitation Limits**: The maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user.
58. **Range**: The difference between the minimum and the maximum of a set of values.
59. **Raw Data**: Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted.
60. **Reagent Blank (method reagent blank)**: A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps.
61. **Reference Material**: A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.
62. **Reference Standard**: A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived.

63. **Requirement**: A translation of the needs into a set of individual quantified or descriptive specifications for the characteristics of an entity in order to enable its realization and examination.
64. **Selectivity**: The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances.
65. **Sensitivity**: The capability of a test method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.
66. **Shall**: Denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification so long as the requirement is fulfilled.
67. **Should**: Denotes a guideline or recommendation whenever noncompliance with the specification is permissible.
68. **Standard Operating Procedures (SOPs)**: A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.
69. **Spike**: A known mass of target analyte added to a blank sample or subsample; used to determine recovery efficiency or for other quality control purposes.
70. **Standard Reference Material (SRM)**: A certified reference material produced by the U.S. National Institute of Standards and Technology and characterized for absolute content, independent of analytical test method.
71. **Supervisor (however named)**: The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance / quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses.
72. **Surrogate**: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.
73. **Test**: A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate.
74. **Test Method**: Defined technical procedure for performing a test.
75. **Testing Laboratory**: Laboratory that performs tests.
76. **Tolerance Chart**: A chart in which the plotted quality control data is assessed via a tolerance level (e.g. +1-10% of a mean) based on the precision level judged acceptable to meet overall quality/data use requirements instead of a statistical acceptance criteria (e.g. +1-3 sigma).

77. **Traceability**: The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons.
78. **Verification**: Confirmation by examination and provision of evidence that specified requirements have been met.

Quality Assurance Policy

Prepared by: _____ Reviewed and Implemented by: _____
Laboratory Director General Manager

The quality assurance policy of Premier Laboratory, Inc. is expressed in the following extract from the Mission Statement in the Premier Laboratory, Inc. Statement of Qualifications.

Premier Laboratory, Inc. provides its customers with high quality analytical reports and services. We distinguish ourselves by working with our customers to define their needs and to meet or exceed their expectations.

Our employees are driven by customer satisfaction. We are knowledgeable of our areas of expertise and continually uphold the stringent protocols required by regulations.

We consistently provide timely results in easy to use formats. We produce superior reports at costs that bring the greatest value to our customers. We strive to make our customers want to use our services and we know that every customer is a tremendous source of referral business to our company.

Each employee is also required to read and sign, and return copy of the Premier Laboratory, Inc. Employee Handbook acknowledgement form to the personnel department. The Employee handbook reiterates Premier Laboratory, Inc.'s mission statement, conflict of interest policy, along with the following Quality Policy statement.

Premier Laboratory, Inc. provides the highest level of quality and service to its customers. Premier Laboratory, Inc. serves its customers by analyzing various matrices and providing legally defensible reports that accurately reflect the results of those analyses. It is the policy of the company that no one falsifies, orders others to falsify the data contained in our reports or to alter, produce, or change any report or information that has not been produced by established laboratory protocol or procedures. If any events cast doubt on the validity of the data we produce, we will notify all effected clients within 24 hours of our awareness of the events.

Violation of this policy is grounds for immediate dismissal without recourse.

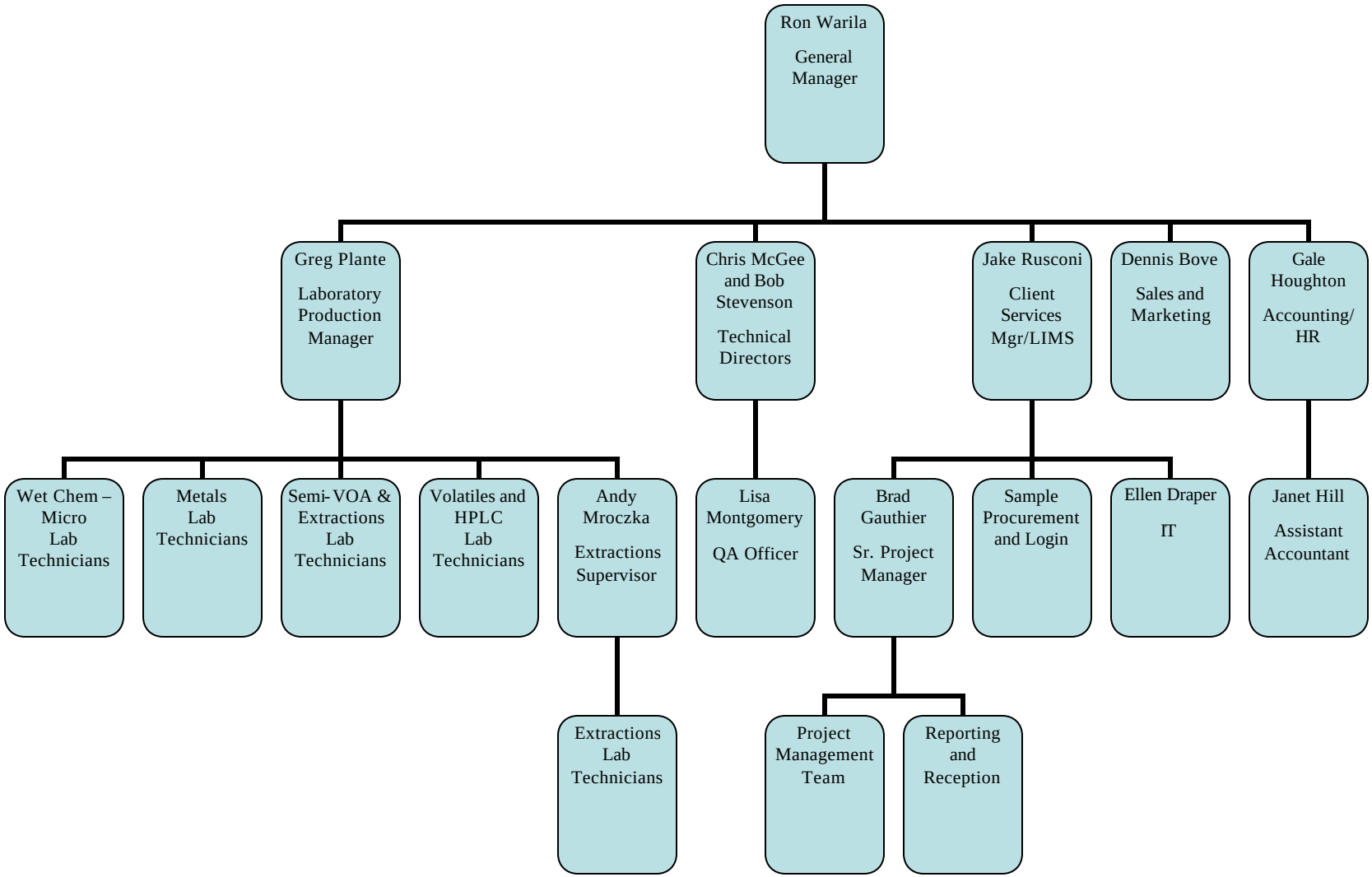
Quality Assurance Objectives

Prepared by: _____ Reviewed and Implemented by: _____
Laboratory Director General Manager

Results generated by environmental laboratory analyses are used to make decisions involving the expenditure of large amounts of time and money, and could even lead to litigious action involving responsible parties. It is imperative, therefore, that the data supplied by the laboratory are of known and measurable quality. The following quality assurance objectives ensure that data produced by Premier Laboratory, Inc. will meet these requirements.

- Ensure compliance with certification requirements
- Ensure compliance with appropriate regulatory agencies
- Ensure compliance with exact contract requirements
- Ensure compliance with published methodologies
- Establish minimum standards consistent with industry practices

The procedures in this manual were developed to achieve these objectives and are binding on all Premier Laboratory, Inc. employees.


Ron Warila
General Manager
Greg Plante
Laboratory Production Manager
Chris McGee and Bob Stevenson
Technical Directors
Jake Rusconi
Client Services Mgr/LIMS
Dennis Bove
Sales and Marketing
Gale Houghton
Accounting/ HR
Wet Chem – Micro Lab Technicians
Metals Lab Technicians
Semi-VOA & Extractions Lab Technicians
Volatiles and HPLC Lab Technicians
Andy Mroczka
Extractions Supervisor
Lisa Montgomery
QA Officer
Brad Gauthier
Sr. Project Manager
Sample Procurement and Login
Ellen Draper
IT
Janet Hill
Assistant Accountant
Extractions Lab Technicians
Project Management Team
Reporting and Reception

Employee Orientation and Training Program

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

Human Resources

1. Employee Handbook Distribution and Discussion
2. State and Federal Forms Completed
3. Insurance Plan Enrolment Form Completed
4. Time Clock Usage Demonstration
5. Laboratory Tour and Employee Introduction

Chemical Hygiene and Laboratory Safety (Performed by the Safety Officer)

1. Chemical Hygiene Orientation and Training Checklist Distribution
2. Chemical Hygiene Plan Distribution and Discussion
3. Right to Know Discussion
    a. Company Policy
    b. Hazard Identification
    c. MSDS Discussion
    d. Exposure and Accident Prevention
    e. Emergency Response
4. Lab Coat and Safety Glasses Distribution
5. Safety Equipment Identification and Demonstration
6. Online Safety Training Course Completed

QA Policy (Performed by the QAO)

1. QA Policy has been read and discussed with QAO
2. Acknowledgement form signed

Ethics Training (Performed by the QAO)

1. Ethics Policy has been read and discussed with QAO
2. Acknowledgement form signed

Laboratory training program outline **(refer to Laboratory Personnel Training section)**

1. New Employees:
    a. The new employee is given access to Standard Operating Procedures (SOP), instrument manuals, and technical literature.

b. The new employee is teamed with an experienced analyst that will oversee the training process.
c. Department manager discusses analytical procedures and QA/QC with the analyst-in-training.
d. The analyst-in-training must demonstrate proficiency by analysis of Initial Demonstration and Capability (IDC) studies.
e. When the analyst-in-training has demonstrated proficiency according to established QA/QC protocols, documentation of proficiency is entered into his/her training folder.

2. Current Employees: (new placement training)

a. The initial training (prior to IDCs being performed) is to be conducted on an established schedule of training sessions each workday for individuals being promoted internally to instrumental analysis or new methods while performing their current work assignments.
b. This training period will last for 2 to 3 weeks and cover the basics of daily operation under the mentorship of the department lead or a fully trained senior analyst.
c. At the conclusion of the training and the filing of acceptable IDCs with the QA Office, the analyst will be deemed certified to perform analysis in the methodology and instrumentation covered during this initial training program.
d. Ongoing training will be given throughout the tenure of all analysts with the goal that each analyst becomes expert in their field of analysis.

3. Current Employees: (re-training in areas of deficiency)

a. The analyst will be paired off with a senior analyst or technician at the direction of the department manager in cooperation with the QA director.
b. The training will include any deficiency found during an audit, review or otherwise. The analyst or technician will only perform the analysis of client samples during the retraining period under the direct supervision of the senior analyst or department manager.
c. The training period will extend until the department manager is satisfied that the training has effectively reeducated the analyst in the proper techniques required to perform the analysis or task. The analyst or technician will be required to perform IDCs at the conclusion of the training where applicable.
d. A written record of the training should be placed either the personnel training records, internal audit files, or other suitable permanent record related to the deficiency occurrence.
e. If discovery came during a formal internal audit, the internal audit follow-up review must be performed no more than 30 days following the retraining to ensure that all procedures remain in practice.

Instrument Specifications

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

I. Pre-purchase Requirements for All Instruments

The Laboratory Director shall review all proposed instrument purchases to ensure the following requirements are met:

a. The instrument must meet all requirements of the analytical procedure(s) for which it will be used.

b. The instrument must meet all requirements of this section of the Quality Systems Manual.

c. The proposed purchase shall be reviewed for compatibility with existing and proposed hardware and software, operator training requirements, and fit into the overall Premier Laboratory, Inc. business plan.

If the purchase includes software, or involves interfacing with software, the proposal must be reviewed by the information systems manager for compatibility and stability.

II. Minimum Requirements for Ancillary Equipment

a. Analytical balances must have a minimum sensitivity of 0.1 mg (0.0001 g) with a precision of ± 1%.

b. General-purpose balances must have a minimum sensitivity less than 1% of the target weight, or, 0.1 g, whichever is less, and with a precision of ± 1%.

c. Visual/ultraviolet spectrophotometers must have a bandwidth of no more than 20 nm and a wavelength accuracy of ± 2.5 nm.

d. pH meters must have an accuracy of at least ± 0.05 pH units and a readability of ± 0.01 pH units within the pH range of 2.0 to 10.0

e. Specific ion meters must have an accuracy and readability of at least ± 5 mV.

f. Electrodes for conductivity meters should have platinum electrodes; non-platinum electrodes must be calibrated against a platinum electrode every six months.

Support Equipment

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

I. Analytical Support Equipment

- Balances
- Ovens
- Refrigerators & Freezers
- Incubators
- Water baths
- Thermometers
- Volumetric dispensing devices

II. Pre-Purchase Requirements for All Support Equipment

The Laboratory Director shall review all proposed instrument purchases to ensure the following requirements are met:

1. The instrument must meet all requirements of the analytical procedure(s) for which it will be used.
2. The instrument must meet all requirements of this section of the Quality Systems Manual.
3. The proposed purchase shall be reviewed for compatibility with existing and proposed hardware and software, operator training requirements, and fit into the overall Premier Laboratory, Inc. business plan.

If the purchase includes software, or involves interfacing with software, the proposal must be reviewed by the information systems manager for compatibility and stability.

All support equipment and devices not meeting required specifications will be removed from service until such time as repairs and/or calibrations are performed to bring the support device back into control.

Purchasing

Prepared by: _____ Reviewed and Implemented by: _____
Laboratory Director General Manager

I. Scope and Applicability

The following procedures must be used when purchasing laboratory supplies.

II. Procedure

1. Each week, all department managers will review the supplies utilized in their departments.
2. The department managers will review the current preferred vendors list for the required item(s) and generate a purchase order (PO). Considerations must be made to ensure that the quantity order will provide an adequate source for the lab with minimal post expiration waste.
3. The department managers will place an order to the appropriate vendor, ensuring that the correct volume and quality of product is ordered.
4. The receiving department will receive all packages shipped to the laboratory and notify the appropriate manager. The manager will check all packing slips to ensure that the correct products have been received and there are no damaged products.
5. The manager will distribute the product(s) to each lead chemist in their respective areas.
6. The chemists will log the product(s) into the standards/reagents tracking module, which will assign a control number to all of the standards/reagents the laboratory receives. The product(s) are then delivered to the appropriate end users.

Balances

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

I. Installation

All balances must be mounted on heavy, shock resistant tables or balance pads.

All balances must be located away from drafts, sources of vibration, and high traffic.

II. Use and Maintenance

1. Check the balance level before use and adjust if necessary.
2. Check the balance pan for cleanliness; clean with a soft-hair brush if necessary.
3. Check the balance tare and adjust if necessary.
4. Each day the balance is used it must be checked with a combination of the following Class 1 weights that cover the range of intended used for each balance: 0.1 g, 10.0 g and 50 g. These checks must be recorded in a balance logbook. Acceptable precision is ±1% of the true value of each weight. Corrective action for balances found to be outside of the acceptable limits are as follows:

    a. Rerun the previous steps of maintenance 1 through 3 above.

    b. Verify that the balance pan is correctly positioned.

    c. Eliminate any outside influences such as vibrations or air turbulence that may be affecting the balance performance.

    d. Reweigh the Class 1 weights.

5. If the balance continues to perform outside the acceptable limits, remove the balance from service and inform the QA office of the need for recalibration immediately.
6. Do not use corrosive chemicals on or around the analytical balance.
7. Allow the material to be weighed to equilibrate to room temperature in a desiccator before weighing.
8. Close all balance doors before recording the weight.
9. Clean the balance thoroughly and close all balance doors after each use.

III. Calibration

All analytical balances are calibrated and cleaned annually by a competent ISO 9001 certified balance service. Service dates are documented on each individual balance.

All Class 1 weights are sent out annually to a NIST certified metrologist for calibration and certification. All NIST Class 1 weight calibration records are maintained in the laboratory.

Refrigerators and Freezers

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

I. Temperature Requirements and Monitoring

Thermometers used for monitoring must be calibrated throughout the acceptable temperature range for the unit that is monitored.

Refrigerator temperatures must be maintained between 1 °C and 4.5 °C. Any thermometer used for monitoring must be graduated in increments no larger than 0.5 °C.

Freezer temperatures must be maintained between -10 °C and -20 °C.

Fill a clean, dry 40-mL vial to the bottom of the threads with glycerin. Place the vial in the refrigerator or freezer at least 6 inches from the door. Position the vial so that the temperature can be taken without manipulating the vial. ***Do not place the vial on the door shelves.*** At a distance from the vial of less than 6 inches, aim the IR thermometer at the vial and take one reading, and then an additional reading to confirm. Record the confirmed temperature reading in the logbook.

Optionally, a thermometer may be added to the vial as a spot check of the temperature throughout the workday. If indications are that the temperature may be out of range, a calibrated measurement must be performed that will dictate any corrective actions. The recorded temperature must not be taken at any time by means of an un-calibrated thermometer. All thermometers that are calibrated are tagged or otherwise labeled and the calibration is current (less than one year past).

A temperature log is maintained in each department. If the unit is used for a special purpose and has an acceptable range different from those above, note this on the temperature log.

The temperature is read and recorded daily for each unit (twice daily for the microbiology department). Adjust the temperature according to the correction factor and record the adjusted temperature and initial the temperature log. If the temperature is drifting close to the acceptance limits, adjust the unit and record the action taken on the temperature log. If the unit is out of range, notify the laboratory manager immediately. Record all corrective action on the temperature log.

II. Maintenance

1. Keep the refrigerator clean at all times.
2. Periodically remove frost buildup.

III. Monitoring Thermometer Calibration

The monitoring thermometer must be calibrated annually. Infrared temperature devices used must be calibrated against a NIST thermometer semi-annually.

1. Calibration:
   a. Remove the cap and thermometer from the vial of glycerin.
   b. Replace the thermometer in the vial without the cap and place a certified thermometer in the vial.
   c. Return the vial to the refrigerator and allow the temperature to equilibrate for at least 1 hour.
   d. The temperature of the certified thermometer must be within the acceptable range before continuing.
   e. Infrared devices are to be calibrated by placing a NIST thermometer in a vial of glycerin, allow the NIST thermometer to stabilize, take 3 consecutive readings with the infrared device and calculate any correction factor based on the average of the 3 readings.
   f. Infrared devices must be checked once per day against a calibrated thermometer. The temperature of the vial containing the calibrated thermometer is measured using the IR gun. This reading is then compared to the temperature shown on the calibrated thermometer. These two readings must agree within 0.5°C, otherwise the IR gun must be recalibrated.
2. Record the following in the temperature-monitoring log:
   a. Analyst (initials)
   b. Date and time of temperature reading
   c. Monitoring Thermometer Temperature (Reg. Therm. Temp., after equilibration)
   d. Unit No. (refrigerator or freezer number)
3. The following calibration information is maintained on file:
   a. Certified Thermometer No.
   b. Thermometer Unit ID (thermometer number)
   c. Date of calibration
   d. Certified Thermometer Temperature (after calibration)
   e. Correction Factor (Reg. Therm. Temp.-Cert. Therm. Temp.)
4. Tag the monitoring meter and record the following on the tag:
   a. Thermometer number
   b. Date of Calibration
   c. Correction factor

Ovens

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

I. Temperature requirements and monitoring

Thermometers used for monitoring must be calibrated throughout the acceptable range for the unit that is monitored. Current IR guns may not be capable of reading within the applicable ranges required, and if so should not be used.

Glassware drying ovens must be maintained at a temperature no higher than 105°C. All other ovens must be maintained within the temperature range specified by the applicable SOP.

Fill a clean, dry container at least 8 cm deep with sand. Insert the thermometer into the sand so that the bottom of the thermometer is 1 - 2 cm above the bottom of the container.

Place the container and thermometer on the center of the top shelf so that the thermometer protrudes through the opening in the top of the oven. Adjust the height of the container so that the acceptable temperature range is visible.

Label the temperature log with the oven number and the acceptable temperature range. If the unit is used for a special purpose and has an acceptable range different from those above, note this on the temperature log.

Read and record the temperature daily for each unit. Adjust the temperature according to the correction factor, record the adjusted temperature, and initial the temperature log. If the temperature is drifting close to the acceptance limits, adjust the unit and record the action taken on the temperature log. If the unit is out of range, notify the laboratory manager immediately. Record all corrective action in the temperature log.

II. Maintenance

Oven

Keep the oven clean at all times.

Monitoring Thermometer

The monitoring thermometer must be calibrated annually.

1. Place a certified thermometer in the container of sand with the monitoring thermometer.
2. Return the vial to the oven and allow the temperature to equilibrate for at least 1 hour.
3. The temperature of the certified thermometer must be within the acceptable range before continuing.
4. Record the following in the temperature-monitoring log:

a. Analyst (initials)
b. Date and time of temperature reading
c. Monitoring Thermometer Temperature (Reg. Therm. Temp., after equilibration)
d. Unit Number (Oven number)

5. The following calibration information is maintained on file:
   a. Certified Thermometer No.
   b. Thermometer Unit ID (thermometer number)
   c. Date of calibration
   d. Certified Thermometer Temperature (after calibration)
   e. Correction Factor (Reg. Therm. Temp.-Cert. Therm. Temp.)
6. Tag the monitoring meter and record the following on the tag:
   a. Thermometer number
   b. Date of calibration
   c. Correction factor

Incubators and Water Baths

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

I. Temperature requirements and monitoring

Thermometers used for monitoring must be calibrated throughout the acceptable temperature range for the unit that is monitored.

- BOD incubators must maintain a temperature of 20.0 °C ± 1.0 °C. Temperatures are monitored and recorded daily.
- Total coliform incubators must maintain a temperature of 35.0 °C ± 0.5 °C. Temperatures are monitored and recorded twice daily.
- Fecal coliform incubators must maintain a temperature of 44.5 °C ± 0.2 °C and a relative humidity of at least 90%. Temperatures are monitored and recorded twice daily.

Fill a clean, dry 40-mL vial to the bottom of the threads with glycerin. Place the vial in the refrigerator or freezer at least 6 inches from the door. Position the vial so that the temperature can be taken without manipulating the vial. ***Do not place the vial on the door shelves.*** At a distance from the vial of less than 6 inches, aim the IR thermometer at the vial and take one reading, and then an additional reading to confirm. Record the confirmed temperature reading in the logbook.

Optionally, a thermometer may be added to the vial as a spot check of the temperature throughout the workday. If indications are that the temperature may be out of range, a calibrated measurement must be performed that will dictate any corrective actions. The recorded temperature must not be taken at any time by means of an un-calibrated thermometer. All thermometers that are calibrated are tagged or otherwise labeled and the calibration is current (less than one year past).

A temperature log is maintained in each department. If the unit is used for a special purpose and has an acceptable range different from those above, note this on the temperature log.

The temperature is read and recorded daily for each unit. Adjust the temperature according to the correction factor and record the adjusted temperature and initial the temperature log. If the temperature is drifting close to the acceptance limits, adjust the unit and record the action taken on the temperature log. If the unit is out of range, notify the laboratory manager immediately. Record all corrective action on the temperature log.

II. Maintenance

Keep the incubators and water baths clean at all times.

Thermometers and Dispensing Pipettes

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

I. Thermometers

All thermometers must be of the appropriate immersion type or calibrated IR for the intended use.

Thermometers used for measurement of water sample temperature must be graduated in 0.1 to 1.0 °C increments, depending on the need.

Thermometers used for temperature monitoring of incubators must be graduated in 0.2 °C increments.

Thermometers used for temperature monitoring of refrigerators, and ovens must be graduated in 0.5 °C increments.

All liquid thermometers used in the laboratory for monitoring support equipment shall be calibrated once each year, digital thermometers quarterly and infrared devices every six months against a NIST certified thermometer. All correction factors are recorded and maintained in the laboratory. A spot check on IR guns must be made each day against a NIST calibrated liquid thermometer to ensure that the electronics are functioning properly.

The NIST certified thermometer used to calibrate laboratory thermometers is sent out yearly to a certified metrologist for calibration against NIST calibration standards. All NIST calibration reports are maintained in the laboratory.

II. Mechanical Volumetric Dispensing Devices

All dispensing devices are purchased with support calibration documentation.

All calibrated dispensing devices should be checked for accuracy on a quarterly basis. The calibration or verification must be within the specification required of the application(s) for which the equipment is used.

Dispensing pipettes, which do not meet accuracy specifications, are removed from service and subsequently re-calibrated with a certified NIST standard or a correction factor must be applied. All certification documentation is maintained in the laboratory.

Instrument Maintenance

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

The minimum maintenance requirements for gas chromatography (GC), gas chromatography/mass spectrometry (GC/MS) and inductively coupled argon plasma (ICP) instruments are found in the Standard Operating Procedures for Maintenance. The maintenance requirements listed are general and minimal; any additional maintenance requirements listed in the manufacturers' manuals are also required and shall be included with these minimum requirements in the Premier Laboratory, Inc. instrument operation manual. The supervisor of each department is responsible for scheduling and assigning instrument maintenance.

All scheduled and unscheduled maintenance activities shall be recorded in the instrument maintenance logbook. A separate instrument logbook is required for each instrument. The maintenance logbook must conform to the requirements of the Laboratory Notebook Procedures section of this manual and must be maintained in the same room as the instrument. The following information shall be recorded for each maintenance event:

- Date and time maintenance was initiated
- Triggering event
- Description of maintenance performed
- Date and time maintenance was completed
- Initials of person who performed maintenance
- Initials of supervisor if maintenance was not performed by supervisor

Glassware Specifications

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

- All glassware used for chemical analysis must be manufactured from borosilicate glass unless specified otherwise by the analytical procedure.
- All volumetric glassware must be Class A. Volumetric glassware shall not be exposed to temperatures greater than 105 °C.
- Dangerously chipped, broken, or cracked glassware shall not be used for analysis. Any broken glassware, which can be repaired without affecting accuracy, will be performed by a professional glass blower.
- Before use, glassware must be cleaned according to the Glassware Cleaning procedure in this manual.
- Mohr and similar measuring pipettes shall not be used for chemical analysis.

Glassware Cleaning

Prepared by: _____ Reviewed and Implemented by: _____
Laboratory Director General Manager

Applicability

This procedure is to be used for cleaning all glassware used for sample analysis.

Important Notes

Glassware cleaning personnel must wear their lab coats, aprons, gloves, and safety glasses as required by laboratory safety policies.

Procedure

Inorganic Lab

a. Thoroughly wash the glassware in tap water and phosphate-free detergent until the glassware is free of visible material. If this does not adequately clean the glassware, soak the glass in Chromerge™ solution for one hour.
b. Rinse the glassware at least four times with tap water to remove all detergent.
c. Rinse the glassware with distilled water at least 2 times.
d. Air dry glassware.

Metals Lab

The following cleaning sequence must be used for all glassware that will contact samples being analyzed for trace metals:

a. Detergent wash
b. Tap water rinse
c. 1:1 nitric acid rinse
d. Reagent water rinse (3 reps)
e. 1:1 hydrochloric acid rinse
f. Reagent water rinse (3 Reps)
g. Air dry glassware in a laminar flow hood.

Volatile Organics Lab

a. Detergent wash glassware.
b. Rinse with tap water.
c. Rinse with organic free water.

d. Oven dry at 105 °C for one hour.
e. Rinse glassware with methanol before using.

Semivolatiles Lab

This procedure is to be performed by the analyst just prior to using the glassware for extraction or analysis.

a. Detergent wash glassware.
b. Rinse with tap water.
c. Oven dry at 105°C for one hour and/or rinse the glassware with acetone or methanol.
d. Rinse the glassware with the solvent to be used in the extraction or analysis procedure.

Reagent Specifications

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

Reagent Water

Reagent water used for chemical or microbiological analyses must meet the following specifications. If any parameters are outside control limits the reagent water cannot be used until the reagent water supply is serviced. Bottled reagent water may be used if the laboratory reagent water supply is out of specifications provided the bottled water is tested and found to meet the requirements below.

Reagent Water for Chemical Analyses

1. Conductivity must be measured and recorded daily. The control limit for conductivity is <2 micro mhos/cm at 25.0 °C.
2. pH must be measured and recorded daily. The pH must be between 5.5 and 7.5.
3. Specific chemical contamination is monitored by the analysis of method blanks. The reagent water supply must meet all method-specific requirements for method blank analysis.

Additional Monitoring for Microbiological Analyses

1. Residual chlorine must be analyzed and recorded monthly, and must be not detectable above 0.05 mg/L by an approved EPA method.
2. Total Organic Carbon should be analyzed and recorded monthly and be <1.0mg/L.
3. Ammonia and Organic nitrogen should be analyzed and recorded monthly and be <0.1mg/L.
4. Heterotrophic plate count must be analyzed and recorded monthly, and it must be < 500 colonies/mL.
5. Cd, Cr, Cu, Pb, Ni, and Zn must be analyzed and recorded annually and the concentration of each metal must be less than 50 µg/L, and collectively no greater than 100 µg/L.
6. Bacterial quality (suitability test) must be analyzed and recorded annually, and must be a ratio between 0.8 and 3.0.
7. A use test must be performed annually, with the student's $t \leq 2.78$.

Reagents

1. All inorganic reagents shall be ACS Reagent Grade or equivalent unless the analytical procedure specifies a different grade.

2. All organic reagents used to prepare standards shall be of the highest quality obtainable. Organic reagents used to prepare general reagent solutions shall be free of detectable interferences as demonstrated by the analysis of acceptable method blanks.
3. All organic solvents shall be free of detectable residue as demonstrated by the analysis of acceptable method blanks. For organic analyses, contamination shall not be restricted to target analytes.

Other Supplies

1. Supplies such as filter paper, glass wool, and boiling beads must be free of contamination as demonstrated by the analysis of acceptable method blanks. For organic analyses, contamination shall not be restricted to target analytes.
2. Supplies such as those listed above used for preparation of organic extracts shall be pre-rinsed with the solvent(s) used in the extraction and concentration procedures.
3. All desiccants must contain moisture indicators.

Purchased Reagents
Labeling, Documentation, and Storage

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

Purchased Chemicals and Solutions

The following information is recorded in the Standards Receipt module of LIMS for all received reagent and chemicals:

1. Date Received
2. Receiver Initials
3. Chemical Name
4. Chemical Type
5. Concentration
6. Purity
7. Expiration Date
8. Vendor Name
9. Vendor Lot #

When all the above information is entered into LIMS and the record saved, a unique identification number is generated for that item. A label is then generated that indicates the tracking number, name, concentration, and expiration date of the reagent or chemical. All purchased chemicals and solutions must have this label. If chemicals or solutions are purchased in case quantities, each individual item in the case must have the label identifying it as from the same lot and received on the same day.

As new stock is received, old stock is rotated so that the oldest stock is most accessible (in front or on top) and the newest stock is least accessible (in back or on bottom).

If the manufacturer does not provide an expiration date, the following default dates shall be recorded on the label:

- Volatile organic solutions: one (1) year from the date opened
- Other solutions with organic solvents: one (1) year from the date opened
- Aqueous solutions: one (1) year from the date opened
- Neat chemicals: Five (5) years from the date opened (provided no loss of physical integrity)
- Microbiology chemicals: six (6) months from the date opened

If the manufacturer does not provide the storage requirements, the following storage requirements apply:

- Aqueous solutions and neat chemicals: store at room temperature
- Solutions in organic solvents:
  a. Sealed ampules may be stored at room temperature until opened
  b. All other containers must be stored in a freezer (-15 °C)
  c. Volatile organic solutions shall not be stored with any other solutions

Reagent Stocks Prepared in the Laboratory

The following information shall be recorded in the stock or working standards module of LIMS:

- Date
- Analyst Initials
- Reagent Name
- LIMS Assigned Purchased Reagent Control Number
- Initial Weight / Volume
- Final Volume
- Final Concentration
- LIMS Assigned Purchased Reagent Control Number for Solvent
- Solvent Type

When all the above information is entered into LIMS and the record saved, a unique identification number is generated for that item. A label is then generated that indicates the tracking number, name, concentration, and expiration date of the stock or working standard.

The following expiration times apply to all reagents where the expiration time is not provided by the method:

- Volatile organic solutions: one (1) year
- Other solutions with organic solvents: one (1) year
- Aqueous solutions: one (1) year

All expiration dates must be prior to the expiration of the parent materials expiration.

If the storage requirements are not provided by the method, the following storage requirements apply:

- Solutions with organic solvents shall be stored in a freezer (-15 °C)
- Volatile organic solutions shall not be stored with any other solutions
- Aqueous solutions shall be stored at room temperature

Purchased Standards
Labeling, Documentation, and Storage

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

Purchased Standards

- All certificates and documentation received pertaining to concentration, purity, traceability, etc. must be retained for a minimum of five (5) years.
- The concentrations of uncertified standards must be verified using primary standards or secondary standards that can be traced to primary standards. Record verification data in the purchased standards receipt module of LIMS.

Standards Receipt

- The following information is recorded in the Receipt Standards Tracking module of LIMS for all purchased standards:

  a. Date Received
  b. Receiver Initials
  c. Standard Name
  d. Concentration
  e. Purity
  f. Vendor Name
  g. Vendor Lot #
  h. Expiration Date

- When all the above information is entered into LIMS and the record saved, a unique identification number is generated for that item. A label can then be generated that indicates the tracking number, name, concentration, and expiration date of the standard.

Labeling

- The following information shall be recorded on the storage container label for each standard as applicable:

  a. Date Received
  b. Standard Name
  c. Concentration
  d. Date of Expiration
  e. LIMS Assigned Control Number

Documentation of Standards Preparation

- The following information shall be recorded in the Stock Standard section of the Standards Tracking module of LIMS:

  a. Date Prepared
  b. Analyst Initials
  c. Stock Standard Name
  d. Initial Concentration of Stock Standard
  e. LIMS Assigned Purchased Standard Control Number
  f. Weight / Vol. of Solute
  g. Final Volume
  h. Solvent Type
  i. LIMS Assigned Purchased Reagent Control Number for Solvent
  j. Final Concentration

When all the above information is entered into LIMS and the record saved, a unique identification number is generated for that item. A label is then generated that indicates the tracking number, name, concentration, and expiration date of the standard.

- The following information shall be recorded in the Working Standard module of LIMS:

  a. Date
  b. Analyst Initials
  c. Working Standard Name
  d. LIMS Assigned Stock Standard Control Number
  e. Stock Standard Concentration
  f. Initial Amount of Stock Standard Used
  g. Final Volume of Working Standard
  h. Working Standard Solvent Type
  i. LIMS Assigned Purchased Reagent Control Number for Solvent
  j. Final Concentration of Working Standard

Labeling

- The following information shall be recorded on the storage container label for each standard as applicable:

  a. Date Received
  b. Standard Name
  c. Concentration
  d. Date of Expiration
  e. LIMS Assigned Control Number

Storage

- The following expiration times apply where the manufacturer or method does not provide the expiration time:

a. Volatile organic standards: one (1) year
b. Other standards with organic solvents: one (1) year
c. Aqueous standards: one (1) year

- The following storage conditions apply where the manufacturer or method does not specify the storage conditions:

a. Standards with organic solvents shall be stored in a freezer (-15 °C)
b. Volatile organic standards shall not be stored with any other standards
c. Aqueous metals standards and stable inorganic standards shall be stored at room temperature.
d. Unstable aqueous standards shall be stored in a refrigerator (1 - 4 °C)

Batch Standards

- These standards are prepared daily or as part of the method preparation batch and shall be recorded as follows:

a. The stock standard solution used to prepare or spike the batch standards must be entered in LIMS as described above.

b. The LIMS tracking number and concentration of the stock standard solution must be recorded in the appropriate method or instrument notebook.

c. For each batch standard, the final concentration and final volume, if different than that of the samples, must be recorded in the appropriate method or instrument notebook, along with the amount of stock standard solution used to prepare that standard.

Personnel Requirements by Function

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

The personnel requirements of this section use the USEPA minimum requirements for certification as a guideline. The requirements are listed by function. If an individual performs more than one function, that individual must satisfy the requirements for all the functions that he or she performs. In addition, many contracts require redundancy for most functions.

This is a guidance document and the requirements herein are not mandatory for Premier Laboratory, Inc. However, all personnel employed by Premier Laboratory, Inc. must meet the minimum requirements for the states in which the laboratory holds or seeks certification, as well as the requirements of any contracts in which the laboratory is engaged.

All employees are given a job description to read and sign. The job description clearly defines the role of each employee of the Premier Laboratory, Inc. team. A copy of the job description and all training material is kept with the employee's personal file.

1. Environmental Laboratory Director

a. Education

- Bachelor's degree in chemistry, biology or other closely related discipline, with at least 24 credit hours in chemistry

b. Experience

- Seven years of experience in an environmental laboratory

2. Quality Assurance Officer

a. Education

- Bachelor's degree in chemistry, biology or other closely related discipline

b. Experience

- Two years of laboratory experience
- One year of applied experience with quality assurance principles and practices in an environmental laboratory

3. Laboratory Manager/Supervisor

a. Education

- Bachelor's degree in chemistry, biology or other closely related discipline, and 30 credit hours of chemistry

b. Experience

- Two years of experience in the environmental analysis of representative inorganic, organic or biological analytes for which the laboratory seeks or maintains certification.
- A master's or doctoral degree in one of the above disciplines may be substituted for one year of experience.

4. Sample Custodian

a. Education

- Bachelor's degree in any scientific or engineering discipline

b. Experience

- One year of experience in sample receiving, log-in, chain-of-custody documentation, and internal transfer
- One year of related supervisory experience.

5. GC/MS Department Lead

a. Education

- Bachelor's degree in any scientific or engineering discipline
- If the degree is not in chemistry, chemistry courses equivalent to a minor in chemistry is required
- A formal training course in GC/MS operation

b. Experience

- Three years of experience in interpretation of GC/MS data, and operation and maintenance of GC/MS systems
- One year of supervisory experience

6. Mass Spectral Interpretation Specialist

a. Education

- Bachelor's degree in any scientific or engineering discipline
- A formal training course in mass spectral interpretation

b. Experience

- Two years of experience in mass spectral interpretation

7. GC/MS Operator

a. Education

- Bachelor's degree in any scientific or engineering disciplines, or increase the experience requirement to three years
- A formal training course in GC/MS operation

b. Experience

- One year of experience in operation and maintenance of GC/MS systems

8. GC Department Lead

a. Education

- Bachelor's degree in any scientific or engineering discipline
- If the degree is not in chemistry, chemistry courses equivalent to a minor in chemistry is required

b. Experience

- Three years of experience in interpretation of GC data, and operation and maintenance of GC systems
- One year of supervisory experience

9. Pesticide Residue Specialist

a. Education

- Bachelor's degree in any scientific or engineering discipline

b. Experience

- Two years of experience in interpretation of GC data, and operation and maintenance of GC systems

10. GC Operator

a. Education

- Bachelor's degree in any scientific or engineering discipline, or increase the experience requirement to three years

- If the degree is not in chemistry, chemistry courses equivalent to a minor in chemistry is required

b. Experience

- One year of experience in operation and maintenance of GC systems

11. Organic Extraction Department Lead

a. Education

- Bachelor's degree in any scientific or engineering discipline

b. Experience

- Three years of experience in organic sample preparation
- One year of supervisory experience

12. Extraction/Concentration Specialist

a. Education

- Associate's degree in any scientific or engineering discipline or a high school diploma in addition to the minimum experience requirements.
- A college level course in general chemistry

b. Experience

- One year of experience in extraction and concentration

13. Inorganic Chemistry Supervisor

a. Education

- Bachelor's degree in any scientific or engineering discipline

b. Experience

- Ten years of laboratory experience
- One year of supervisory experience

14. ICP Spectroscopist

a. Education

- Bachelor's degree in any scientific or engineering discipline
- Specialized training in ICP spectroscopy

b. Experience

- Two years of experience with ICP analysis of environmental samples

15. ICP Operator

a. Education

- Bachelor's degree in any scientific or engineering disciplines or increases the experience requirement to four years
- A short course in ICP

b. Experience

- One year of experience in operation and maintenance of ICP systems

16. Inorganic Sample Preparation Specialist

a. Education

- Associate's degree in any scientific or engineering discipline or a high school diploma in addition to the minimum experience requirements.
- A college level course in general chemistry

b. Experience

- Six months of experience in an analytical laboratory
- If microwave digestion is used, six months of experience in sample dissolution using microwave digestion techniques is required

17. Classical Chemistry Technician/Specialist

a. Education

- Bachelor's degree in any scientific or engineering disciplines or increase the experience requirement to three years

b. Experience

- One year of experience in classical procedures

18. Microbiology Supervisor

a. Education

- Bachelor's degree in science
- A minimum of four credits in general microbiology

- A minimum of two weeks formal training in microbiological analysis of drinking water

b. Experience

- One year of experience in microbiology

19. Microbiology Specialist

a. Education

- Bachelor's degree in any scientific or engineering disciplines or increase the experience requirement to four years, (an Associate's Degree may be substituted for 2 years of experience).

b. Experience

- One year of experience in microbiology

20. Systems Manager

a. Education

- Bachelor's degree with four or more intermediate courses in programming, information management, database systems management, or systems requirements analysis

b. Experience

- Three years of experience in data systems management or programming
- One year of experience with the software being used for data management and generation of laboratory reports

21. Programmer Analyst

a. Education

- Bachelor's degree with four or more intermediate courses in programming, information management, database systems management, or systems requirements analysis

b. Experience

- Two years of experience in systems or applications programming
- One year of experience with the software being used for data management and generation of laboratory reports

Premier Laboratory, Inc. Ethics Policy

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

Premier Laboratory, Inc. Ethics Policy Form:

Premier Laboratory, Inc. Ethics Policy

Premier Laboratory, Inc. provides the highest levels of quality and service to its customers. Premier Laboratory, Inc. serves its customers by analyzing various matrices and providing legally defensible reports that accurately reflect the results of those analyses. It is the policy of the company that no one falsifies or orders others to falsify the data contained in our reports, or to alter, produce, or change any report or information that has not been produced by established laboratory protocols or procedures.

Confidentiality is an important aspect of your employment at Premier Laboratory, Inc. Premier Laboratory, Inc. accepts work from its clients on a private and confidential basis. In the course of your work, you may acquire information that is of a confidential nature regarding Premier Laboratory, Inc. or its clients. All employees receive a copy of the Conflict of Interest and Confidentiality Policy in the Corporate Handbook. You are bound to its conditions of confidentiality, and agree not to disclose any confidential information learned about Premier Laboratory, Inc, or its clients to any person or entity, except as may be necessary in the course of your duties while employed at Premier Laboratory, Inc.

It is understood and agreed that breach of the policies contained herein will be grounds for immediate termination of employment.

Employee:_____ Date:_____

Witness:_____ Date:_____

Premier Laboratory, Inc. requires that all employees are formally introduced to the ethical and legal aspects of the generation and reporting of data at all levels throughout the laboratory. A discussion with either the Laboratory Director, QA Officer, or both is conducted with each employee during orientation and a copy of the form below is completed for the employee's permanent file. Special notice must be made to the following in regards to maintaining the data integrity of all work and practices performed:

- Emphasis is put on the importance of conducting all laboratory work under the ethical practices prescribed by the laboratory in this statement and to question any practice performed or observed that leaves any doubt to its ethical nature. All observances must be reported to the analyst's supervisor immediately and without hesitation.
- Only staff that has agreed to and signed the ethics statement is permitted to work in the laboratory.
- The QA Officer (QAO) will act as the data integrity advisor and will provide a receptive environment and complete confidentiality when dealing with any ethical dilemmas brought to light by an employee.
- All records of an incident will be kept in confidential files and maintained for 10 years.
- Reports and data generated in the laboratory will be randomly reviewed by the QAO with the purpose of verifying that data integrity has been maintained according to the policies of the laboratory. This review may also include the introduction of double blind samples into the workflow as real samples to gauge performance of the staff.

Laboratory Personnel Training

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

I. The manager of administration shall orient all new employees and provide each new employee with an Employee Packet. The new employee shall verify that he or she has reviewed the materials provided in the Employee Packet by signing the form provided at the end of the Employee Handbook. Each employee is presented with a job description stating all expected duties and requirements for the position. All employees must acknowledge the receipt of their job description by signing the document. An original copy of the employee's job description is kept in his/her personal file in the administrative department.

II. Safety training is the responsibility of the laboratory safety officer.

1. Each employee whose duties involve work in the laboratory or sample management areas shall receive a copy of the laboratory contingency plan, chemical hygiene plan, and chemical waste plan. These must be read, and the employee must sign a statement indicating the plans were understood.
2. The employee shall be given a tour of the laboratory, and all safety equipment and exit locations shall be pointed out.
3. In addition to the above, employees whose duties require access to the hazardous waste storage room shall receive respirator training and a respirator fit test.

III. Each new analyst shall receive orientation from his or her immediate supervisor. This orientation shall include location of the Quality Manual, SOPs, notebooks, and physical layout of the department. The new analyst shall be briefed on quality assurance practices, use of SOPs, and laboratory etiquette.

IV. All analysts have access to all SOPs, Quality Manuals, Chemical Hygiene Plans, Waste Disposal Plans, and Premier Laboratory, Inc.'s Technical Library. All employees are encouraged to attend technical seminars, read technical literature, and to extend their quest for knowledge beyond Premier Laboratory, Inc.. All pertinent information on method modifications, industry changes, and regulatory changes are presented to department managers for dissemination to all personnel.

V. Each analyst must be qualified in each analysis he or she is to perform.

a. The analyst will be provided with a copy of the Premier Laboratory, Inc. SOP for the procedure in which he or she is to be qualified. The analyst will be provided an opportunity to discuss the procedure with the department manager or a lead technician designated by the department manager. When a SOP has been read and understood by the employee, the new employee will sign the appropriate sections of the Analysis

Training Program Training Record form. The new employee will then observe the specific analysis being performed by an experienced analyst. This will allow the new employee to witness the exact execution of the procedure and allow for direct question and answer between the new employee and the experienced analysts. When it is determined by the department manager that an analyst is ready to perform an analysis, an experienced analyst will be assigned to observe technique and maintain the quality assurance of the SOP throughout the entire procedure. Upon completion of the training session, the department manager will query the analyst about the SOP. The department manager will then sign the appropriate sections of the Analysis Training Program Training Record indicating that the analyst has been trained and understands the SOP.

b. The employee will then proceed with an initial demonstration of capability by performing the specific analysis with quality control samples. Upon successful completion of the initial demonstration of capability, the department manager will complete an Initial Demonstration of Capability Certification Statement for the analyst. The completed IDC form will be maintained in the employee-training file located in the Quality Assurance Office.

c. All new employees are provided with the Premier Laboratory, Inc. Quality Manual when hired. It is the responsibility of each employee to read and comprehend the Quality Manual. When the quality assurance officer is satisfied that an employee understands the contents of the Quality Manual, the employee and the quality assurance officer will sign the acknowledgement documentation form, located in the Quality Assurance Office.

d. All analysts will perform on an annual basis at least one of the following:

- The analysis of at least one (single or double blind) quality control standard for each analysis the analyst has been assigned to perform after training has been completed. The analysis is method-based; therefore the QCS may include only compounds necessary to demonstrate proficiency. Successful analysis of a blind performance sample on a similar test method using the same technology (e.g., GC/MS volatiles by purge and trap for methods 524.2, 624 or 8260) would only require documentation for one of the test methods.
- Participation in a formal proficiency study in the applicable methods, with passing scores.
- Another demonstration of capability;
  - At least four consecutive laboratory control samples with acceptable levels of precision and accuracy;
  - Alternatively, analysis of authentic samples that have been analyzed by another trained analyst with statistically indistinguishable results.

Approved Analytical Methods

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

Most environmental regulations stipulate which methods must be used to perform parameter specific analyses. This stipulation may take the form of a specific procedure, or a list of procedures or references from which an appropriate method may be chosen. In general, these procedures must be performed without modification. There are two exceptions:

1. If a regulatory agency modifies a published procedure, the modified procedure must be used for analyses performed within the agency's jurisdiction.
2. The stipulated procedure may not be appropriate for analysis of some samples. (This most often happens when a water method is specified for a non-aqueous sample.) When this occurs, the laboratory must, with the client's permission, work with the regulatory agency to determine a course of action. Possible actions are, in order of preference, to (1) use a different procedure, (2) use an agency modification, (3) use a laboratory modification, or (4) delete the analysis for the affected samples. When contacting the regulatory agency, the laboratory should be prepared to suggest appropriate alternate or modified procedures.

   Note: Any agreement reached between the laboratory, client, and regulatory agency must be confirmed in writing, and the written document must include the scope of the agreement. (The scope of the agreement may be specific samples only, a specific project, any project for this agency, etc.) The written confirmation must be included in all applicable hard copy data packages.

Premier Laboratory, Inc. is restricted to methods from the following list. When selecting methods from this list, the laboratory must ensure that the method is appropriate for the regulation and the sample to be analyzed.

Leachate Procedures

1. TCLP Bulk Extraction by SW-846 method 1311
2. TCLP Zero Headspace Extraction by SW-846 method 1311
3. SPLP Extraction by SW 846 method 1312

Organic Extractions

1. Extraction Procedures
   a. Pesticides and PCBs by SW-846 method 3510C
   b. Pesticides and PCBs by SW-846 method 3520C
   c. Pesticides and PCBs by SW-846 method 3550B
   d. Pesticides and PCBs by SW-846 method 3580A
   e. Semivolatiles (BNA) by SW-846 method 3510C
   f. Semivolatiles (BNA) by SW-846 method 3520C
   g. Semivolatiles (BNA) by SW-846 method 3550B
   h. Semivolatiles (BNA) by SW-846 method 3580A
2. Cleanup Procedures
   a. Acid Cleanup by 3665A
   b. Alumina Cleanup by 3611B
   c. Florisil Cleanup by EPA method 608/SW-846 method 3620B
   d. Silica Gel Cleanup by 3630C
   e. Sulfur Cleanup by EPA method 608/SW-846 method 3660B

Organic Analyses

1. **Gas Chromatography Methods**
   a. HAAs in Drinking Water by EPA method 552.2
   b. Herbicides by EPA method 515.3
   c. Herbicides by SW-846 method 8151A
   d. Pesticides by EPA method 505
   e. Pesticides and PCBs by EPA method 608
   f. Pesticides by SW-846 method 8081A
   g. PCBs (screen) by EPA method 505
   h. PCBs by SW-846 method 8082
   i. Petroleum Hydrocarbons Fingerprint by SW-846 method 8015M
   j. Petroleum Hydrocarbons by 8100M
   k. Semivolatiles by EPA method 504.1
   l. Semivolatiles by EPA method 525.2
2. **Gas Chromatography/Mass Spectrometry Methods**
   a. Semivolatiles (BNA) by EPA method 625
   b. Semivolatiles (BNA) by SW-846 method 8270C
   c. Volatiles by EPA method 524.2
   d. Volatiles by EPA method 624

e. Volatiles by SW-846 method 8260B
f. EPH/VPH by MADEP EPH/MADEP VPH
g. Endothall by EPA method 548.1

3. **HLPC**
   a. Carbamates by EPA method 531.2
   b. Glyphosate by EPA method 547
   c. Diquat by EPA method 549.2

Metals Preparation and Analyses

1. **Digestion Procedures**
   a. Acid Digestion of Aqueous Samples for Total Metals by method 3010A
   b. Acid Digestion of Sediments, Sludges, and Soils by method 3050B

2. **ICP Methods**
   a. General Metals by EPA method 200.7
   b. General Metals by SW-846 method 6010B
   c. General Metals by EPA method 200.8
   d. General Metals by SW-846 method 6020

3. **Cold Vapor Methods**
   a. Mercury by EPA method 245.2
   b. Mercury by SW-846 method 7470A
   c. Mercury by SW-846 method 7471A

Classical Chemistry

Methods for Water and Aqueous Preparations

1. Acidity by EPA method 305.1 / SM20 method 2310B
2. Alkalinity by SM20 method 2320B
3. Alkalinity, Bicarbonate, by SM16 method 403
4. Ammonia Nitrogen by EPA method 350.1 / SM20 method 4500NH_3-G
5. Ammonia Nitrogen with Distillation by EPA method 350.2 / SM20 method 4500NH_3-G
6. Biochemical Oxygen Demand (BOD), 5-Day, by SM20 method 5210B
7. Biochemical Oxygen Demand (BOD), 20-Day, by SM20 method 5210B
8. Biochemical Oxygen Demand, Carbonaceous (CBOD), 5-Day, by SM20 method 5210B
9. Biochemical Oxygen Demand, Carbonaceous (CBOD), 20-Day, by SM20 method 5210B
10. Biochemical Oxygen Demand, Nitrogenous (NBOD), 5-Day, by SM20 method 5210B
11. Biochemical Oxygen Demand, Nitrogenous (NBOD), 20-Day, by SM20 method 5210B
12. Chemical Oxygen Demand (COD) by HACH method 8000 / SM20 method 5220D

13. Chloride by EPA method 325.2, method 300/ 9251/ SM20 method 4500Cl-D
14. Chlorine Demand by SM20 method 4500Cl-G/ 2350B
15. Chlorine, Residual, by Hach 8021/ SM20 method 4500Cl-G
16. Chromium, Hexavalent, by Hach 8023 / SM20 method 3500Cr-D
17. Color by SM20 method 2120B
18. Conductance, Specific, by SW-846 method 9050/ SM20 method 2510B
19. Cyanide, Free, by EPA method 335.4 / SM20 method 4500CN-E
20. Cyanide, Amenable, by SM20 method 4500CN-G
21. Cyanide, Total by EPA method 335.4 /SW-846 method 9012A / SM20 method 4500CN-E
22. Fluoride by EPA method 340.2, method 300 / SM20 method 4500F-C
23. Hardness by SM20 method 2340B
24. Kjeldahl Nitrogen, Total (TKN) by EPA method 351.2
25. Nitrate Nitrogen by EPA method 300
26. Nitrite Nitrogen by EPA method 300
27. Nitrate Nitrogen by Calculation (nitrate-nitrite *minus* nitrite)/ SM20 method 4500NO_3-F
28. Nitrate-Nitrite Nitrogen by SM20 method 4500NO_3-F
29. Nitrite Nitrogen by EPA method 354.1 / SM20 method 4500NO_3-F
30. Odor (TON) by SM20 method 2150B
31. Oil & Grease by EPA method 1664A
32. Organic Nitrogen by Calculation (TKN *minus* Ammonia-N) / SM20 4500NO_3-F
33. Orthophosphate Phosphorus by EPA method 365.1, method 300 / SM20 method 4500P-F
34. Oxygen, Dissolved (DO) by EPA method 360.1/Hach 8229/ SM20 method 4500O-G
35. pH by SW-846 method 9040B, SM4500H^+-B
36. Phenolics by EPA method 420.1/SW846 9065
37. Phosphorus by EPA method 365.1 / SM20 method 4500P-F
38. Salinity by SM16 method 210
39. Solids, Settleable by SM20 method 2540F
40. Solids, Total (TS) by SM20 method 2540B
41. Solids, Total Dissolved (TDS) by SM20 method 2540C
42. Solids, Total Suspended (TSS) by SM20 method 2540D
43. Solids, Total Volatile (TVS) by EPA method 160.4 / SM20 method 2540E
44. Solids, Total Volatile Suspended (TVSS) by SM20 2540D,E
45. Sulfate by Hach 8051 / EPA 300 / SM20 method 4500SO_4^{2-}-E, SM15 426C
46. Sulfide by EPA method 376.2 / SW-846 method 9030 / SM20 method 4500S^{2-}-D
47. Sulfite by EPA method 377.1/ Hach 8071/ SM20 method 4500SO_3^2-B
48. Surfactants (MBAS) by SM20 method 5540C
49. Tannin by Hach 8193
50. TOC by SM5310C
51. Turbidity by EPA method 180.1/ SM20 method 2130B
52. Solids, Total (TS) by 209F / SM20 method 2540G
53. Specific Gravity by SM16 method 213E

54. Ignitability by SW-846 method 1010
55. Paint Filter Liquids Test by SW-846 method 9095A

Microbiology

1. Coliforms, Fecal (MTF) by SM20 method 9221E
2. Coliforms, Total (MF) by SM20 method 9222B
3. Coliforms, Total (MF) by EPA method 1604
4. E. coli (modified mTEC) by SM20 method 9221E
5. E. coli (MF) by EPA method 1604
6. Enterococci (MF) by EPA 1600
7. Heterotrophic Plate Count by SM20method 9215B
8. Microscopic Identification (Algae Scan) / SM19 method 10200F

Waste Characterization

The methods in this category are specific to waste characterization analyses. Methods in other categories may also be required for complete characterization. **CAUTION: Care must be exercised when analyzing samples for waste characterization. Wear appropriate safety gear and perform all analyses in an approved fume hood.**

a. Bulk Density
b. Cyanide Spot Test
c. Flammability
d. Hexane Solubility
e. Odor
f. Oxidizer Spot Test
g. Peroxide Spot Test
h. pH
i. Physical State
j. Redox Potential Spot Test
k. Sulfide Spot Test
l. Viscosity
m. Water Solubility/Reactivity

Method Detection Limits

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

The method detection limit for a procedure is the smallest concentration of analyte that can be measured with known confidence. This procedure is designed for applicability to a wide variety of sample types ranging from reagent (blank) water containing analyte to wastewater containing analyte. The following procedure for calculating method detection limits is derived from 40 CFR 136 Appendix B. Method detection limits must be determined before analysis for any method can begin. Method detection limits are repeated annually or whenever a major change to the method occurs.

Method Detection Limit (MDL)

The MDL procedure that follows is taken from 40 CFR 136 Appendix B. This procedure is defined as "the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero". The results obtained are specific to the sample matrix and analytical system.

1. Make an estimate of the detection limit using one of the following:
   a. The concentration value that corresponds to an instrument signal/noise in the range of 2.5 to 5.
   b. The concentration equivalent of three times the standard deviation of replicate instrumental measurements of the analyte in reagent waster.
   c. That region of the standard curve where there is a significant change in sensitivity, i.e., a break in the slope of the standard curve.
   d. Instrumental limitations. It is recognized that the experience of the analyst is important to this process.
2. Prepare reagent (blank) water that is as free of analyte as possible. Reagent or interference free water is defined as a water sample in which analyte and interfering concentrations are not detected at the method detection limit of each analyte of interest. Interferences are defined as systematic errors in the measured analytical signal of an established procedure caused by the presence of interfering species (interfering). The interfering concentrations presupposed to be normally distributed in representative samples of a given matrix.
3. a. If the MDL is to be determined in reagent (blank) water, prepare a laboratory standard (analyte in reagent water) at a concentration which is at least equal to or in the same concentration range as the estimated method detection limit (recommended between 1 and 5 times the estimated method detection limit). Proceed to Step 4.

b. If the MDL is to be determined in another sample matrix, analyze the sample. If the measured level of the analyte is in the recommended range of one to five times the estimated detection limit, proceed to Step 4.

If the measured level of analyte is less than the estimated detection limit, add a known amount of analyte to bring the level of analyte between one and five times the estimated detection limit. If the measured level of analyte is greater than five times the estimated detection limit, there are two options.

1. Obtain another sample with a lower level of analyte in the same matrix if possible.
2. The sample may be used as is for determining the method detection limit if the analyte level does not exceeds 10 times the MDL of the analyte in reagent water. The variance of the analytical method changes as the analyte concentration increases from the MDL, hence the MDL determination under these circumstances may not truly reflect method variance at lower analyte concentrations.

4. Take a minimum of seven aliquots of the sample to be used to calculate the method detection limit and process each through the entire analytical method. Make all computations according to the defined method with final results in the method reporting units. If a blank measurement is required to calculate the measured level of analyte, obtain a separate blank measurement for each sample aliquot analyzed. The average blank measurement is subtracted from the respective sample measurements.

   It may be economically and technically desirable to evaluate the estimated method detection limit before proceeding. This will:

   (1) prevent repeating this entire procedure when the costs of analyses are high, and
   (2) ensure that the procedure is being conducted at the correct concentration. It is quite possible that an inflated MDL will be calculated from data obtained at many times the real MDL even though the level of analyte is less than five times the calculated method detection limit. To insure that the estimate of the method detection limit is a good estimate, it is necessary to determine that a lower concentration of analyte will not result in a significantly lower method detection limit. Take two aliquots of the sample to be used to calculate the method detection limit and process each through the entire method, including blank measurements as described above. Evaluate these data:

5. If these measurements indicate the sample is in desirable range for determination of the MDL, take five additional aliquots and proceed. Use all seven measurements for calculation of the MDL.

   If these measurements indicate the sample is not in correct range, re-estimate the MDL, obtain new sample as in Step 3 and repeat Step 4.

6. Use the equations from Appendix A of this manual to calculate the standard deviation, then calculate the MDL as follows:

$$\mathbf{MDL = 3.143\ s}$$

where: MDL = method detection limit in the same units as sample concentrations
s = standard deviation of 7 analyte concentration measurements

Report results to the same number of significant digits as used to report sample concentrations.

7. Calculate the upper control limit at 95% confidence from two or more replicates as follows:

$$UCL = k\sqrt{\frac{\sum s_i^2}{n}}$$

where: UCL = upper control limit calculated from MDL study data
k = factor from Table 1 corresponding to the number of replicates
s_i = standard deviation of 7 analyte concentration measurements from study i
n = number of replicates

Report results to the same number of significant digits as used to report sample concentrations.

Table 1: Students' *t* Values at the 99% Confidence Level

Number of Replicates	Degrees of Freedom (n-1)	$t_{(cn-1,.99)}$
7	6	3.143
8	7	2.998
9	8	2.896
10	9	2.821
11	10	2.764
16	15	2.602
21	20	2.528
26	25	2.485
31	30	2.457
61	60	2.390

The values of k for table were calculated as the Student's t value at 99% confidence times the chi square over degrees of freedom a
95% confidence.

Initial Demonstration of Capability

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

The initial demonstration of capability is used primarily to preclude a laboratory from analyzing unknown samples via a new, unfamiliar method prior to obtaining some experience with it. It is expected that as laboratory personnel gain experience with this method the quality of data will improve beyond those required here.

An initial demonstration of method performance must be made prior to using any test method, and at any time there is a significant change in instrument type, personnel, or test method. All initial demonstrations of capability are documented on the appropriate forms and maintained in each employee's file.

The following procedure will be used to perform an initial demonstration of capability.

1. A quality control sample is obtained from an outside source.
2. The quality control sample is diluted in a blank (i.e.: methanol, 10% nitric acid, distilled water, etc.) at least 10 times the method stated or laboratory-calculated detection limits, ideally to a mid-calibration level. Four aliquots of the QC sample are created.
3. The four aliquots are analyzed according to the test method specified.
4. Using the four results, the average recovery and standard deviation of the population are calculated for each parameter of interest.
5. For each parameter, the average recovery and standard deviation are compared to the corresponding acceptance criteria for precision and accuracy in the test method and/or the laboratory generated acceptance criteria. If average recovery and the standard deviation meet the acceptance criteria, the analysis of actual samples may begin. If any one of the parameters exceeds the acceptance range, the performance is unacceptable for that parameter.
6. If a parameter fails acceptance, the analysts must locate and correct the source of the problem and rerun the IDC.
7. An example of the IDC form is shown on the following page.

Initial Demonstration of Capability
Certification Statement

Laboratory Name: **Premier Laboratory, Inc.**

Laboratory Address: **61 Louisa Viens Dr.; Dayville, CT 06241**

Date: _____

Analyst(s) Name(s): _____

Matrix: _____

Parameter: _____

Method number: _____

We, the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method, which is in use at this facility for the analysis of samples under the National Environmental Laboratory Accreditation Program, has met the Initial Demonstration of Capability.
2. The test method was performed by the analyst identified on this certification.
3. A copy of the test method and the laboratory-specific SOPs are available on-site for all personnel.
4. The data associated with the initial demonstration capability are true, accurate, complete and self-explanatory [(1)].
5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized inspectors.

Supervisor	Signature	Date
Quality Assurance Officer	Signature	Date

This certification form must be completed each time an initial demonstration of capability study is completed.

[(1)] True: Consistent with supporting data.
Accurate: Based on good laboratory practices consistent with sound scientific principles/practices.
Complete: Includes the results of all of the supporting performance testing.
Self-explanatory: Data properly labeled and stored so that the results are clear and require no additional explanation.

Preparation, Review, Revision, and Distribution of SOPs

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

The procedures in this section provide the means by which standard operating procedures (SOPs) are prepared, reviewed, revised, approved, distributed, maintained, and archived. In the text that follows, "SOP" refers to a document describing a single procedure, such as this procedure, and "SOP manual" or "manual" refers to a collection of related procedures within a single binding, such as the Quality Systems Manual.

Preparation

1. The following three items must be included as the first pages to appear in each SOP manual.
   a. The title page shall contain the title of the SOP manual and the signature of the person(s) responsible for overview of the activities addressed in the manual.
   b. The Distribution Control Page shall contain a brief description of the document control policy and a statement declaring whether the copy is controlled. For controlled copies, the following additional information shall be entered in black ink:
      - Controlled copy number
      - Name and affiliation of person to whom the manual is issued
      - Signature of the Quality Assurance Officer
      - Expiration date
   c. The Authorized Signatories Page shall contain the signatures of approval from the Quality Assurance Officer, Laboratory Director, Department Supervisor, and a Managing Member of the Company.
   d. The table of contents shall contain the method, title, latest revision date, and page or document number for each SOP included in the manual. The table of contents serves as the revision control document and therefore must be revised for every revision of the manual.
2. Each page of the SOP must have a document control header in the upper right corner of each page with the following four lines.
   a. Title of SOP manual
   b. Method number
   c. Revision number and Date
   d. Effective Date
3. The following items must appear immediately following the document control header on the first page of each SOP:
   a. Title of the SOP

b. Signature of the person with primary responsibility for writing the SOP ("Prepared by")
c. Signature of the person with primary responsibility for overview of the activities addressed by the SOP, if different from the writer (Reviewed and approved by)
d. Signature of the person directly responsible for implementing the procedure, if different from those above (Reviewed and implemented by)
e. A footnote indicating the next date of review/revision

4. The remainder of each SOP will vary depending on its function. The following subsections present minimum standards for each type of SOP.

   a. References: Include the full name, volume, edition, publication date, and method number for each reference.
   b. Applicability: List the target analytes, matrices, and regulations for which the method is applicable.
   c. Important Notes: Information, warnings, and cautions that should be known to anyone performing the procedure.
   d. Procedure: Step-by-step instructions for performing the procedure.
   e. Quality Control: List all quality control requirements for method validation, instrument performance check, initial and continuing calibration, method blanks, duplicate analyses, matrix spike analyses, laboratory control sample analyses, and all method-specific quality control, including control limits and corrective action.
   f. Calculations: Formulae for all calculations required to obtain reported values, in the correct units.
   g. Reagents: List all neat chemicals with specifications; list solutions with detailed and specific preparation instructions. Include storage conditions and expiration times for all solutions.
   h. All employees will acknowledge that they have read and understood the individual SOPs that pertain to their area of work by signing the method acknowledgment logbook or SOP revision logbook in the QA Office.

Review, Revision, and Approval

1. Each SOP or, sections subject to revision, will be opened for comments to those who are familiar with its content and/or those who will use it. All proposed SOP revisions must be submitted to the QAO for review and subsequent distribution.

2. After review of the comments and suggested modifications of the procedure, the SOP in its revised form will be reviewed and approved by the Laboratory Director, QA Officer, Department Lead/Supervisor, and person(s) responsible for overview of the activities addressed by the SOP. A packet containing a hard copy of the SOP with accepted revisions in place and the original text lined out will be distributed to all relevant individuals and supervisors along with a SOP Revision Acknowledgement sheet. Once each person has read and comprehended the SOP, they are then required to sign and date the SOP Revision Acknowledgement sheet attached. The packet will then be archived in the QA Office in the SOP Revisions Log. The QAO shall revise the table of contents and the revised SOP will replace the current electronic SOP. A master hardcopy will be produced and stored by the

QAO. The obsolete electronic table of contents and SOP will be marked revised and subsequently moved to archival storage at the time of the review/reprinting.

Distribution and Maintenance

1. Hardcopy SOP manuals are distributed as controlled documents for project specific external (out of lab) distribution or uncontrolled documents for external review. The distribution of controlled documents must be recorded to ensure that they are updated when new or revised SOPs are released.
2. SOP manuals for use within Premier Laboratory, Inc. are available as uncontrolled hardcopies or may be viewed as controlled electronic documents on the laboratory network.
3. SOP manuals submitted to regulatory agencies in support of certification or submitted to clients for contract compliance must be controlled for the duration of the certification or contract. SOPs are reviewed on an annual basis.
4. The Quality Assurance Officer (QAO) shall be responsible for maintaining updated masters for all documents.

   a. The QAO shall maintain a distribution log for all controlled documents in which the following information shall be recorded for each controlled copy:

      - Document name
      - Controlled copy number
      - Name of recipient
      - Date of issue
      - Date control expires (for copies with contract duration)

   b. Obsolete hardcopies of all SOPs must be archived for at least ten years from the date they are removed from circulation.

Laboratory Notebook Procedures

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

Laboratory Notebooks

1. Laboratory notebooks shall be permanently bound with each page sequentially pre-numbered.
2. All notebook entries must be in black ink.
3. Correct errors by drawing a single line through the incorrect data. Initial and date the error, and continue with the correct data. **No other means of correcting errors is permitted.**
4. Record the analytical method number at the top of each page. Do not include more than one method per page.
5. Record all information needed to reconstruct the analyses and recalculate results.
6. Do not record confidential information such as client names, project names, *etc.*, in the laboratory notebook.
7. Each page must be signed and dated by the analyst and his or her supervisor when analyses are completed. Any unused portion of the page must be lined out.

Laboratory Notebook Control

1. A logbook of issued laboratory notebooks shall be maintained and kept on file with the current logbook templates. This log shall be maintained in a bound, pre-numbered notebook meeting all the requirements of a laboratory notebook.
2. The following information shall be recorded in the logbook when each notebook is issued:
    a. notebook number
    b. number of previous notebook
    c. signature of person receiving the notebook
    d. notebook use (instrument log, solids analysis, etc.)
3. Upon completion of each issued laboratory notebook, it must be placed in archival storage. Laboratory notebooks must be archived for at least ten years following the date of the last entry.

Multi-Level Data Review and Record Retention

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

The most diligent care in performing an analysis is lost if the data is not processed properly. Every calculation or manipulation performed on the data must be independently validated to prevent this loss. The department managers review all raw data and for completeness and consistency. When data is reviewed and verified by department managers, the status of the work order is changed to "verified" in the LIMS. The report generation department then generates the final report, along with the invoice and routing sheet. The package is then forwarded to the project management department.

The project manager will review the package for completeness compared to the original chain of custody, review the invoice, and forward the package to quality assurance officer or the laboratory director for final review and signature.

The multi-level review of all final report packages ensures that the customer receives the highest level of quality assurance.

All analytical reports, logbooks, raw data, electronic data, and any other related records shall be maintained for a minimum of ten years.

Data Reduction, Validation, and Review

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

Reference

Premier Laboratory, Inc. LIMS Manual: Developed internally for use with Premier Laboratory, Inc.'s BLISS system

Premier Laboratory Statement of Qualifications

Applicability

All data generated within the laboratory is subject to a rigorous process of reduction, validation and review.

Important Notes

Attention to detail is critical in data reduction, validation and data package generation.

Procedure

Stage 1

Raw data is entered into the Laboratory Information Management System (LIMS) in two ways, by either direct acquisition from instrumentation or through analyst input at a computer workstation. The raw data is then compiled by the LIMS system with all further calculation being performed within the LIMS results module (i.e.: % solids applied, conversion of reporting units, application of minimum reporting limits, etc.). The status of the analysis is changed to "completed" in the LIMS at the completion of this stage.

Stage 2

The department managers review all raw data against the final data generated for both accuracy and completeness. Any potential errors in calibration requirements or spike recoveries will be reviewed at this level. When data is reviewed and verified by department managers, the status of the work order is changed to "verified" in the LIMS.

Stage 3

The report generation department then generates the final report, along with the invoice and routing sheet. The package is then forwarded to the project management department. The project manager will review the package for completeness compared to the original chain of custody, review the invoice.

Stage 4

The package is forwarded to one of the laboratory directors for final review and signature. If the sample results exceed any established maximum contaminant levels or the results indicate a potentially harmful situation; the client is to be notified within 24 hours of validation of the data.

Stage 5

If a data package is required, the package is forwarded to the Quality Assurance Officer. The QAO will note which department(s), if any, will be required to provide quality control documentation. Upon completion of the data package, a final review by the QAO will take place before the package is sent to the client.

The multiple levels of review of all final report packages ensure the customer receives the highest level of quality assurance.

Method Validation

Prepared by: _____ Reviewed and Implemented by: _____
Laboratory Director General Manager

A method validation study must be performed before any new procedure can be used for sample analysis. This section delineates the minimum requirements for a method validation study. Most analytical procedures have specific requirements with respect to method validation, which are found in the method reference. If the method-specific requirements are more restrictive than these requirements, they supersede these requirements; otherwise, they are in addition to these requirements.

1. Perform a method detection limit (MDL) study following the procedures in the Method Detection Limits section of this manual. The following acceptance criteria must be met:
    a. The MDL determined must be less than the concentration used for the study but not less than 20% of the concentration used for the study.
    b. The MDL study must demonstrate the ability to achieve the detection limits published in the reference within the experimental variance of the method. If the experimental variance is not given in the reference, the MDL determined by the study cannot be more than 50% greater than the published detection limit. If the detection limit is not given in the reference, contact the Laboratory Director or Quality Assurance Officer for appropriate action.
    c. MDL studies are repeated whenever a major change is instituted in a current method.
2. Perform an initial demonstration of capability following the procedures in the Initial Demonstration of Capability section of this manual.
    a. The initial demonstration of capability must meet the quality control limits published in the method and/or meet the quality control limits established by the laboratory.
    b. The initial demonstration of capability is repeated for each analyst performing the test.

Instrument Performance Check

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

Instrument performance checks ensure the proper functioning of an analytical instrument prior to analysis. Specific performance check procedures are included in the instrument operation manuals and are frequently introduced into analytical procedures. When an instrument has passed the required performance check(s), no adjustment of operating parameters is permitted that will alter instrument performance. In addition to the frequency requirements of the instrument operation manuals and analytical methods, an instrument performance check must be performed whenever the operating parameters have been changed.

If an instrument fails the performance check requirements, corrective action must be initiated. If the instrument manual and analytical method have conflicting requirements, the requirements of the method shall have precedence over the operation manual requirements.

Corrective action will vary from instrument to instrument. The following general procedure should be followed to find and correct the problem.

1. Review the instrument adjustment procedure and ascertain that the instrument is properly adjusted.
2. If adjustment fails to solve the problem, review the trouble-shooting procedures to determine the cause of the problem.
3. If the problem cannot be solved by adjustment and trouble-shooting, the instrument must be adjusted or repaired by a qualified professional service representative.

After adjustments and/or repairs are completed, the instrument performance check must be repeated. The instrument may not be used for analysis until the performance check has passed. It is the responsibility of the department manager to oversee corrective action. Documentation of corrective action is recorded in the sample run log. Subsequent acceptable performance checks are also documented in the sample run log.

Initial Calibration

Prepared by: _____ Reviewed and Implemented by: _____
Laboratory Director General Manager

An initial calibration must be performed before any other analyses may be performed. The initial calibration establishes the relationship between instrument response and sample concentration, and the working concentration range of the analytical system. This section delineates the minimum requirements for initial calibration analysis.

Most analytical procedures have specific requirements with respect to calibration that are found in the method SOP. If the method-specific requirements are more restrictive than these requirements, they supersede these requirements; otherwise, they are in addition to these requirements.

1. **Levels:** A minimum of five (5) non-zero concentrations must be used unless the method specifies otherwise. The lowest concentration must be at the quantitation limit. The highest concentration should be near but below the upper linear range of the instrument. The remaining three concentrations must be evenly spaced between the lowest and highest concentrations.

2. **Calibration:** Instruments are calibrated by performing the calculations from the equations found in each specific method. The resulting equation is also used to calculate sample concentrations.

3. **Frequency:** The initial calibration must be performed whenever a continuing calibration fails to meet quality control limits.

4. **Quality Control Limit:** The correlation coefficient for each target analyte must be greater than 0.995. An initial calibration has failed quality control requirements and corrective action must be initiated if the correlation coefficient for any target analyte is less than 0.995.

5. **Verification:** The initial calibration must be verified by analyzing an independently prepared standard with target analyte concentrations within the calibration range. The standard is obtained from a separate vendor or, from a different lot number if the same vendor is used. This standard must pass all the continuing calibration check requirements. Some verification standards may have manufacturer's certified acceptance range that is narrower than that established by the method. If these standards are analyzed as received, then the manufacturer's certified range of acceptability will be followed, along with the certified true values.

6. **Corrective Action:** If an initial calibration fails quality control requirements, the analytical system must be investigated to determine the cause of the problem and an acceptable initial calibration must be obtained. It is the responsibility of the analyst to correct deficiencies. Documentation of corrective action is recorded in the sample run log. Subsequent acceptable performance checks are also documented in the sample run log.

Continuing Calibration Check

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

A continuing calibration check must be performed before any quality control or sample analyses may be performed. The continuing calibration check determines whether the initial calibration is still valid. This section delineates the minimum requirements for continuing calibration check analysis.

Most analytical procedures have specific requirements with respect to calibration that are found in the SOP. If the method-specific requirements are more restrictive than these requirements, they supersede these requirements; otherwise, they are in addition to these requirements.

1. **Level:** The concentration of the continuing calibration check standard should be the same as the mid-level initial calibration standard.

2. **Procedure:** Calculate the concentration of each analyte of interest in the continuing calibration check standard from the equations derived from the initial calibration. Refer to each method for approved quantification technique and equations.

3. **Frequency:** The continuing calibration check must be performed before sample analyses (except immediately following an initial calibration). The sample batch has a time limit of 24 hours. Additional continuing calibration checks throughout the sequence may be required for specific methods.

   Note: An analytical sequence begins with the initial calibration or continuing calibration check and continues without interruption until the final continuing calibration check or until the 24 hour clock expires. No changes in analytical conditions are permitted during an analytical sequence.

4. **Quality Control Limits:** The calculated analyte concentrations must fall within the laboratory established or method required theoretical (or true) values. A continuing calibration check has failed quality control requirements and corrective action must be initiated if the concentration of any target analyte falls outside the established theoretical values.

5. **Corrective Action:**

   a. If the continuing calibration check that starts an analytical sequence fails quality control requirements, the continuing calibration check may be repeated. All target analytes must pass quality control requirements in the second continuing calibration check in order for samples to be analyzed; otherwise, a new initial calibration must be performed.

b. If any continuing calibration check during or at the end of an analytical sequence fails quality control requirements, analyses must be terminated, the problem resolved, and all analyses performed after the failed checks must be repeated. It is the responsibility of the analyst to correct deficiencies. Documentation of corrective action is recorded in the sample run log. Subsequent acceptable performance checks are also documented in the sample run log.

Method Blank Analysis

Prepared by: _____ Reviewed and Implemented by: _____
Laboratory Director General Manager

Analytical systems must be demonstrated to be free of contamination by the successful analysis of method blanks. This section delineates the minimum requirements for method blank analysis. Many analytical procedures have specific requirements with respect to method blank analysis, which are found in the SOP. If the method-specific requirements are more restrictive than these requirements, they supersede these requirements; otherwise, they are in addition to these requirements. **Method blanks are not to be used to correct analytical results**.

1. **Preparation:** Method blanks must be prepared at a frequency of one per batch of samples per matrix type per sample extraction or preparation method. They are analyzed in the same manner as the samples with which they are prepared and analyzed, except that sample is omitted or, for analysis of water samples, replaced with deionized water. The final concentration of all reagents used is the same in the method blank and the samples.
2. **Quality Control Limits:**
   a. The blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated sample batch and
   b. The blank contamination exceeds the concentration present in the samples and is greater than 1/10 of the specified regulatory limit.
   c. The exempted target analytes are:
      - methylene chloride
      - acetone
      - 2-butanone
      - toluene
      - bis(2-ethylhexyl) phthalate
3. **Corrective Action:** If a contaminated method blank is encountered, sample analysis must be terminated and the analytical system must be investigated to determine the cause of the problem. If the method blank contamination is such that it cannot be corrected, the method blank and all associated samples (including quality control samples) must be prepared again and reanalyzed. If the method blank contamination is such that re-extraction is not possible, all associated samples are flagged with the appropriate data qualifiers. It is the responsibility of the analyst to correct deficiencies. Documentation of corrective action is recorded in the sample run log. Subsequent acceptable performance checks are also documented in the sample run log.

Sample Duplicate Analysis

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

Sample duplicates are analyzed to demonstrate the precision of an analytical system. This section delineates the minimum requirements for sample duplicate analysis.

Many analytical procedures have specific requirements with respect to sample duplicate analysis that are found in the SOP. If the method-specific requirements are more restrictive than these requirements, they supersede these requirements; otherwise, they are in addition to these requirements.

1. **Preparation:** For inorganic analyses, two equivalent aliquots of the sample selected for duplicate analysis are prepared and carried through the preparation and analysis procedures together. For organic analyses that do not specify a duplicate analysis, a second matrix spike is prepared and carried through the preparation and analysis. All references to sample duplicates in this document apply equally to matrix spike duplicates.
2. **Frequency:** A sample duplicate shall be prepared and analyzed at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation method. This frequency is for a single matrix and method; quality control samples cannot be shared by matrices or methods.
3. **Relative Percent Difference (RPD):** The relative percent difference is used to evaluate precision for analyses that measure concentration of an analyte directly. The relative percent difference **cannot** be calculated if either or both analyses are below the quantitation limit.

$$\mathbf{RPD} = \frac{\mathbf{100\,(\,C_1 - C_2\,)}}{\mathbf{C_{avg}}}$$

where: C_1 = original sample concentration
C_2 = sample duplicate concentration
C_{avg} = average concentration of the two samples

4. **Difference:** The absolute value of the difference of two measurements is used to evaluate precision for analyses that do not measure analyte concentrations directly (e.g., temperature, pH, Ignitability).
The following equation applies:

$$\mathbf{D = |M_1 - M_2|}$$

where: D = absolute value of the difference between two measurements
M_1 = original sample measurement
M_2 = duplicate sample measurement

5. **Control Limits:** The equations for calculating the mean and standard deviation are included in Appendix A of this manual. Use the data from at least 20 sample and sample duplicate analyses to calculate warning and control limits. Eliminate outliers using the method in Appendix A.

    a. **RPD:** The warning limit is the mean RPD plus two times (2x) the standard deviation, and the control limit is the mean plus three times (3x) the standard deviation. A sample duplicate analysis has failed quality control requirements and corrective action must be initiated if (1) the RPD is greater than the control limit or (2) the concentration for one analysis is less than the quantitation limit and the concentration for the other analysis is greater than two times (2x) the quantitation limit.

    b. **Difference:** The warning limit is the mean difference plus two times (2x) the standard deviation, and the control limit is the mean plus three times (3x) the standard deviation. A sample duplicate analysis has failed quality control requirements and corrective action must be initiated if the difference is greater than the upper control limit.

    c. A sample duplicate analysis has failed quality control requirements and corrective action must be initiated if:

        (1) The RPD is greater than the control limit or 20% for the duplicate pair.

        (2) The concentration for one analysis is less than the quantitation limit and the concentration for the other analysis is greater than two times (2x) the quantitation limit.

        (3) The absolute difference is greater than the quantitation limit when the measured concentrations are between the quantitation limit and (2x) the quantitation limit.

6. **Corrective Action:** If a sample duplicate analysis fails quality control requirements, the failure must be included in the non-conformance summary. No further action is required.

Matrix Spike Analysis

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

Matrix spikes are analyzed to demonstrate the accuracy of an analytical system. This section delineates the minimum requirements for matrix spike analysis.

Many analytical procedures have specific requirements with respect to matrix spike analysis that are found in the SOP. If the method-specific requirements are more restrictive than these requirements, they supersede these requirements; otherwise, they are in addition to these requirements.

1. **Preparation:** Matrix spikes are prepared with the same sample size that is used for the samples with which they are analyzed. The spiking solution is added immediately after the sample aliquot is measured, and is mixed as thoroughly as practical with the sample. Subsequent treatment and analysis of the spiked sample is the same as that for the un-spiked samples.
2. **Level:** The amount of spike added to the sample must give a concentration that will fall between the lowest and highest calibration standards when carried through the analysis procedure without dilution. The preferred concentration is 25% of the highest calibration standard; this concentration yields usable recovery data without dilution at low to moderate sample concentrations.
3. **Frequency:** Unless otherwise mandated in the SOP, a matrix spike shall be prepared and analyzed at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available. For organic analyses that do not specify a duplicate analysis, a matrix spike duplicate shall be prepared and analyzed with each matrix spike and shall serve the same function as a duplicate analysis. In instances when inadequate sample volume is available for spiking purposes, an alternate sample with sufficient volume and of a similar matrix must be selected and spiked. This sample may be one that has been previously analyzed and data is available for spike recovery evaluation. However, sample holding times for the method must still be adhered to for the spiked aliquot.
4. **Recovery (Accuracy):**

$$C_T = \frac{A_S}{S_S} \qquad R = \frac{100(C_S - C_U)}{C_T}$$

where: C_T = theoretical concentration of spiked sample
A_S = amount of analyte added to spiked sample
S_S = size of sample aliquot spiked
R = recovery, %
C_S = measured concentration of spiked sample
C_U = measured concentration of unspiked sample

5. **Relative Percent Difference (Precision):** See the section on Sample Duplicate Analyses.

6. **Control Limits:** The equations for calculating the mean and standard deviation are included in Appendix A of this manual.

    a. **Recovery:** Use the data from at least 20 matrix spike analyses to calculate warning and control limits for recovery. Do not include data from matrix spike duplicate analyses. Eliminate outliers using the method in Appendix A.

    The upper warning limit is the mean recovery plus two times (2x) the standard deviation, and the upper control limit is the mean plus three times (3x) the standard deviation. The lower warning limit is the mean recovery minus two times (2x) the standard deviation, and the lower control limit is the mean minus three times (3x) the standard deviation. A matrix spike analysis has failed quality control requirements and corrective action must be initiated if the recovery is greater than the upper control limit or less than the lower control limit.

    b. **RPD:** See the section on Sample Duplicate Analyses.

7. The percent recovery must be plotted on control charts showing the mean, warning limits, and control limits.

8. **Corrective Action:** If the procedure utilizes surrogate spikes in every sample analyzed, all samples for which the surrogate spikes meet quality control requirements shall be considered acceptable regardless of the associated matrix spike recoveries. Samples for which surrogate spikes do not meet quality control requirements must be re-prepared and reanalyzed.

    If the procedure does not utilize surrogate spikes, the following corrective action protocol applies.

    a. If a matrix spike recovery is outside control limits and the RPD is within control limits, the problem is attributed to matrix interference and no further action is required.

    b. If the matrix spike is outside control limits and the laboratory control sample is acceptable, the problem is attributed to matrix interference and no further action is required.

    c. If the matrix spike recovery is outside control limits and the concentration of the same analyte in the unspiked sample is more than four times (4x) the concentration of spike analyte added, the problem is attributed to sample analyte concentration and must be noted in the non-conformance summary. No further action is required.

    d. If the matrix spike recovery is positive and outside control limits and the RPD is outside control limits, the problem must be noted in the non-conformance summary. No further action is required.

    e. If the matrix spike has zero or negative recovery, report the problem to the supervisor or technical director.

9. Matrix spike data is kept with the daily folder for each instrument. Final data packages, which are stored with client report files, also contain spike records.

Laboratory Control Sample Analysis

Prepared by: _____ Reviewed and Implemented by: _____
Laboratory Director General Manager

Laboratory control sample or as otherwise named is analyzed to demonstrate the accuracy of an analytical system. They are supplemental to matrix spikes and, because environmental matrices are not involved, provide useful information when matrix interference is encountered. Laboratory control sample (LCS) analyses are required except in the rare instances when they are specifically not required by the analytical procedure or project. Refer to the SOP for method-specific requirements or project management department.

This section delineates the general requirements for LCS analysis. If the analytical procedure requires LCS analysis, the method-specific requirements supersede these requirements.

1. **Preparation:** Laboratory control samples are prepared in the same manner as matrix spikes except that spiking solution is added to laboratory reagent water (DI water) instead of sample. Subsequent treatment and analysis of the LCS is the same as that for environmental samples.
2. **Level:** The amount of spike added must be the same as that used for matrix spikes unless specified otherwise by the analytical method.
3. **Frequency:** An LCS shall be prepared and analyzed at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction except for analytes for which spiking solutions are not available. Quality control samples cannot be shared by matrices or methods.
4. **Recovery (Accuracy):**

$$C_T = \frac{A_S}{S_S} \qquad R = \frac{100(C_S - C_U)}{C_T}$$

where: C_T = theoretical concentration of spiked sample
A_S = amount of analyte added to spiked sample
S_S = size of sample aliquot spiked
R = recovery, %
C_S = measured concentration of spiked sample
C_U = measured concentration of unspiked sample

5. **Control Limits:** The equations for calculating the mean and standard deviation are included in Appendix A of this manual.
   a. Use the data from at least 20 LCS analyses to calculate warning and control limits for recovery. Eliminate outliers using the method in Appendix A.

b. The upper warning limit is the mean recovery plus two times (2x) the standard deviation, and the upper control limit is the mean plus three times (3x) the standard deviation. The lower warning limit is the mean recovery minus two times (2x) the standard deviation, and the lower control limit is the mean minus three times (3x) the standard deviation. An LCS analysis has failed quality control requirements and corrective action must be initiated if the recovery is greater than the upper control limit or less than the lower control limit.

6. The percent recovery must be plotted on control charts showing the mean, warning limits, and control limits.
7. **Corrective Action:** If the procedure utilizes surrogate spikes in every sample analyzed, all samples for which the surrogate spikes meet quality control requirements shall be considered acceptable regardless of the associated LCS recoveries. Otherwise, if the LCS recovery is outside control limits, sample analysis must be terminated and the analytical system must be investigated to determine the cause of the problem. The LCS and all associated samples (including quality control samples) must be prepared again and reanalyzed.

Quality Assurance Assessment

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

The previous sections in this manual addressed specific quality assurance requirements and their implementation. This section addresses audits, which are the primary procedures for assessing compliance with these requirements and the requirements of other Premier Laboratory, Inc. procedural manuals.

Each audit is based on a single Premier Laboratory, Inc. procedural manual. The auditor shall conduct the audit as directed in the *Instructions to the auditor* near the top of the audit form. Immediately following the audit, the auditor shall meet with the Managing Member or his/her designee and determine appropriate measures to correct any deficiencies found. If a condition is found that affects the accuracy or defensibility of reported data, the deficient practice must be suspended until the condition is corrected. Otherwise, a maximum of 30 days may be allowed to correct deficiencies. The corrective actions to be taken shall be recorded and maintained in the audit files located in the QA/QC Office. A follow-up audit shall be performed within 45 days to determine whether the corrective actions were properly completed.

The Quality Assurance Program Compliance Audit is based on the Quality Manual and determines the degree to which a laboratory complies with general quality assurance program requirements. This audit is performed annually.

The Qualifications Audit is based on the Statement of Qualifications and determines whether quality assurance information is accurate and updated. This audit is performed annually.

The Sample Management Audit is based on the Client Services Manual and determines the degree of compliance with sample management procedures. This audit is performed annually.

The Analytical Method Audit is based on the Standard Operating Procedures for analyses and determines whether analytical procedures are being performed properly. This audit is performed annually.

Proficiency Testing and Internal Audits

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

Premier Laboratory, Inc. completed the final EPA sponsored rounds of WS and WP proficiency testing in 1998. Premier Laboratory, Inc. will comply with state mandated proficiency testing requirements in addition to meeting NELAC specifications for proficiency testing.

Premier Laboratory, Inc. also from time to time measures quality assurance by processing double and single blind quality control samples in the laboratory independent of the proficiency schedules. The QAO is responsible for maintaining the internal quality control program.

The QAO, in cooperation with the department managers, is responsible for performing internal audits, citing deviations from the SOPs, and overseeing corrective action. The quality assurance compliance audit, qualifications audit, and the sample management audit are performed annually. Analytical method audits are ongoing according to a schedule that provides a flexible framework which works inclusive of laboratory operations.

The following audit forms are available in the QA office:

- Quality Assurance Program Compliance Audit
- Qualifications Audit
- Sample Management Audit
- Analytical Method Audit

Duplicate Selection Procedure

Prepared by: _____ Reviewed and Implemented by: _____
Laboratory Director General Manager

Reference:

2003 NELAC Standard, Chapter 5, Appendix D, Page 5D-5

Scope and Applicability

The following procedure is designed as a guide to selecting duplicate samples and duplicate matrix spikes that are representative of the range of matrix, which are encountered throughout the entire client base. All personnel involved in sample preparation and/or analyses are to follow the prescribed procedure. The respective department manager is to be consulted when encountering a new or unusual matrix.

Important Notes

Historical data pertaining to sample origin, client, and recovery should be maintained in a notebook that relates to each area of testing (i.e. organics, metals, microbiology, etc.).

Procedure

The Client Services Department will notify the department managers of new clients or sample sites for established clients prior to the samples being logged into the LIMS system.

The department managers will inform the analysts performing the analysis of the new client/site.

The analyst will incorporate into the sample flow a duplicate sample/duplicate matrix spike chosen at random from the new client/site, or rotate randomly among the routine client/site samples. Additionally, duplicate selection will be made for each matrix (solid, aqueous, solvent, etc.) when applicable to the new client/site work order.

The results for the duplicate recovery will be documented in the permanent records along with any deviations from the established recovery limits as described in the Quality Manual.

Analysts are to notify the department manager of any recovery deviations to be included in a case narrative/non-conformance summary.

Sample Homogenization Procedure

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

Reference:

NELAC Quality Systems Manual, rev. 12, 2001, chapter 5

Scope and Applicability

The following procedure is designed as a guide to homogenizing samples prior to analysis. Where sampling (as in obtaining sample aliquots from a submitted sample) is carried out as part of the test method, the laboratory shall use appropriate techniques to obtain representative sub samples. All personnel involved in sample preparation and/or analyses are to follow the prescribed procedure. The respective department manager is to be consulted when encountering a new or unusual matrix.

Important Notes

Care must be taken to insure that all apparatus used in the homogenization of samples does not introduce contaminates of any kind. All equipment must be cleaned according to the procedures applicable to the area of analysis. Special considerations must be made for samples that are to be analyzed for parameters in multiple areas of the laboratory.

Procedure:

All samples are to be thoroughly mixed in a secondary container if the primary container is at more than 50% capacity.

Samples are to be blended thoroughly in the case of soil, sludge, tars and heavy oils. When samples are composed of materials that require particle size reduction to comply with the analysis methodology, the particle reduction must be performed first, then the sample homogenized. Particle reduction may be performed using reasonable methods so long as the sample is not exposed to undue temperatures or pressures that may alter the composition of the sample.

Samples, which contain various different objects combined such as cloth, solid wood, plastics or other miscellaneous objects, must be handled as follows. The sample components must be weighed to determine each components percent of the total sample. Particle reduction is performed independently on each component to provide adequate sample mass to perform all analysis in duplicate. The sample components are then recombined in proportion to their original percent distribution in the total sample and stored in a new sample container appropriate to the analysis.

The department managers will inform the analysts performing the analysis of any client requests when handling unusual samples that require specific manipulations in order to homogenize properly.

Analysts are to notify the department manager of any deviations to be included in a case narrative/non-conformance summary.

Environmental Monitoring

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

Reference:

2003 NELAC Standard, Chapter 5, Section 5.5.3, page 22

Scope and Applicability

The following procedure is designed to monitor levels of contamination in each area of the laboratory and for each area of testing. The monitoring is conducted by the department managers in cooperation with the Quality Assurance Officer.

Important Notes

Recovery records are to be maintained in a notebook that relates to each area of testing (i.e. organics, metals, microbiology, etc.). The records should include the following elements: collection locations, collection container type, baseline levels, recovery results, investigative findings, and corrective actions.

Procedure

The testing is conducted under the program in cases when significant activity in an area may introduce contamination.

1. Select the appropriate sample containers (glass or plastic) for the area of testing.
2. Place an open collection container at each workstation in which sample containers are routinely exposed to the environment, (i.e. lab benches, storage racks). Locate additional collection containers in areas that may contribute directly to the introduction of contamination, (heating vents, windows or doors).
3. Leave the containers undisturbed for 2-24 hours.
4. Process the collection containers in the same manner as a routine sample, and record any results above the reported detection limits in the areas logbook.

The department manager must be informed of any contamination detected immediately upon discovery.

Corrective Actions

If a positive result is detected for any contaminant, the following corrective actions must be carried out.

1. The level of contamination is to be entered into the logbook for the collection location site and compared with the established baseline levels.
2. The contamination source is to be tentatively identified based on the recovery results.
3. The detected contamination is to be cleaned or otherwise eliminated in the area immediately.
4. A retest of the contaminated site is to be performed to determine the effectiveness of the elimination process.
5. Applicable method blank results should be reviewed to determine if the contamination adversely effected sample analysis.
6. All actions are to be recorded in the monitoring logbook.

Standards and Reagents Tracking

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

Electronic Logbook (Elog)

As of April 2003, all standards and reagents are logged into the Standards Tracking module in LIMS when prepared or received.

Receipt

1. Click on "Receipt" in the left hand column in the Standards Tracking module found in LIMS.
2. All newly received standards and neat chemicals used to make standards are logged into the system by entering the following information into the appropriate fields on the receipt page:
   a. **Name** – enter the common name for the standard or chemical that is listed in the SOP or a unique descriptive name applied universally.
   b. **Conc** (Concentration) – enter the concentration of the standard or reagent, for multi-analyte mixes enter "varies" or likewise. This value is not used in any calculation.
   c. **Received** – enter the date the standard or reagent was received in the laboratory.
   d. **Received by** - enter the initials of the receiver.
   e. **Sealed Expiration** - enter the manufacturer's expiration or if there is none specified enter the default found in this manual.
   f. **Opened Expiration** - enter the date of expiration for after the standard or reagent container seal is broken.
   g. **Opened by** - enter the initials of the first person that breaks the container seal.
   h. **Analyte** – on the title bar, click once on the spreadsheet icon with the blue field, and a pop up box will appear. Select from the list each analyte in the standard or reagent (individually). Click "OK" button. The analyte will appear in the analyte list in the bottom left of the reagent page. Click on the number in the analyte row, and enter the concentration in the highlighted box at the bottom center of the page; in the case of neat chemicals enter the concentration of the species of interest, i.e.: KNO_3 where the atomic mass for K = 39.0983 + N = 14 + O = 3(16) = 101; NO_3 contributes 61 % of the atomic mass of KNO_3. Select the units of measure with the drop down menu.

      If the standard is received on a regular basis, proceed in entering the information below first, and then perform the following sequence for borrowing the analytes from a previously received standard. (The total volume units and department being entered are key to using the borrow function.)

      (1) In the title bar, click on the borrow icon, (one beaker replicating into two beakers). A window with a list will pop up.

(2) Scroll down the left menu and find the previously received standard that is to be borrowed.
(3) Expand the tree (click on the "+" symbol) and the individual analytes will appear with check boxes.
(4) Check the box of the standard or reagent and the individual components will be checked that are to be borrowed.
(5) Click on the move icon in the center of the menus to transfer the analytes selected to the right menu, click "OK".
(6) The analytes will appear in the analyte list on the receipt page.
(7) Enter the concentrations and amounts in the same manner as the individually selected analyte instructions above.

i. **Solvent** (check box)- check if the reagent is a solvent, this will drop prevent the concentration calculation from being performed
j. **Vendor**- enter the manufacturer's name, (not the distributor).
k. **Lot # -** the manufacturer's lot number.
l. **Part # -** the manufacturer's part number.
m. **Purity** – enter the purity, generally applies only to neat chemicals. This value is not used in any calculation.
n. **QC Checked** – the date that the standard or reagent (usually a solvent) is checked for purity and suitability thru analysis
o. **QC Checked by** – the initials of the analyst performing the QC check analysis
p. **Department** – the laboratory department in which the standard or reagent is primarily used.
q. **Location** – the storage location of the standard or reagent
r. **Temperature** – the storage temperature.
s. **Amount** – enter the initial volume or weight of the standard or reagent and select the units from the drop down menu.

3. When all entries are verified for correctness, save by clicking on the floppy disk icon on the title bar. Click on the print icon on the title bar once for each label required. All standards and reagents must be properly labeled. Open dates must be recorded on the label at the time of opening.

Stock Solution

1. Click on "Stock" in the left hand column in the Standards Tracking module found in LIMS.
2. All newly prepared standards made directly from receipt standards and neat chemicals are logged into the system by entering the following information into the appropriate fields on the Stock page.

a. **Name** – enter the common name for the standard or chemical that is listed in the SOP or a unique descriptive name applied universally.
b. **Conc** (Concentration) – enter the concentration of the standard or reagent, for multi-analyte mixes enter "varies" or likewise. This value is not used in any calculation.

c. **Mixed** – enter the date the stock solution is made.
d. **Mixed by** – enter the initials of the analyst preparing the stock solution.
e. **Expiration** –the date will fill in automatically after all of the parent solutions are entered.
f. **Final volume** – enter the final volume of the stock solution being prepared select the units from the drop down menu.
g. **Current** – the current amount available will automatically be entered as the stock is consumed in the preparation of working standards.
h. **Department** – enter the department in which the standard or reagent will be used.
i. **Location** – the storage location of the standard or reagent
j. **Temperature** – the storage temperature.
k. **Discarded** (check box) – check the box when a standard is made but discarded prior to being consumed.
l. **Display Units** – select the units from the drop down menu for which the final solution will be expressed.
m. **Analyte** – on the title bar, click once on the spreadsheet icon with the blue field, and a pop up box will appear. Select from the list each analyte in the standard or reagent (individually). Click [OK] button. The analyte will appear in the analyte list in the bottom left of the stock page. Click on the negative symbol to collapse the tree for each analyte (if expanded). Highlight each analyte individually to activate the Aliquot field and Units drop down menu at the bottom center of the stock page. Enter the amount used in the Aliquot field and select the units of measure with the drop down menu.

3. If the standard is made on a regular basis, proceed in entering the information below first, and then perform the following sequence for borrowing the analytes from a previously made standard. The total volume units and department being entered are key to using the borrow function.

a. In the title bar, click on the borrow icon, (one beaker replicating into two beakers). A window with a list will pop up.
b. Scroll down the left menu and find the previously received standard that is to be borrowed.
c. Check the box next to the stock standard to be borrowed or expand the tree (click on the "+" symbol) and the individual analytes will appear with check boxes.
d. Check the boxes of the analytes that are to be borrowed.
e. Click on the move icon in the center of the menus to transfer the analytes selected to the right menu, click "OK".
f. The analytes will appear in the analyte list on the stock page.
g. Enter the concentrations and amounts in the same manner as the individually selected analyte instructions above.
h. The analyte names and concentrations in the prepared stock solution will appear in the right lower box on the stock page.
i. When all entries are verified for correctness, click the save icon on the title bar.
j. Click on the print icon on the title bar once for each label required. All stock standards must be properly labeled.

Working Solution

1. Click on "Working" in the left hand column in the Standards Tracking module found in LIMS.
2. All newly prepared standards made from stock standards and neat chemicals are logged into the system by entering the required information in the appropriate fields on the Working page.
   a. **Name** – enter the common name for the standard or chemical that is listed in the SOP or a unique descriptive name applied universally.
   b. **Conc** (Concentration) – enter the concentration of the standard or reagent, for multi-analyte mixes enter "varies" or likewise. This value is not used in any calculation.
   c. **Mixed** – enter the date the working solution is made.
   d. **Mixed by** – enter the initials of the analyst preparing the stock solution.
   e. **Expiration** –the date will fill in automatically after all parent solutions are entered, or if the standard is prepared fresh daily check off the daily box to the right of the field. The date and time of expiration will then be automatically entered for a daily standard.
   f. **Final volume** – enter the final volume of the stock solution being prepared select the units from the drop down menu.
   g. **Current** – the current amount available will automatically be entered as the stock is consumed in the preparation of working standards.
   h. **Department** – enter the department in which the standard or reagent will be used.
   i. **Location** – the storage location of the standard or reagent
   j. **Temperature** – the storage temperature.
   k. **Discarded** (check box) – check the box when a standard is made but discarded prior to being consumed.
   l. **Display Units** – select the units from the drop down menu for which the final solution will be expressed.
   m. **Analyte** – on the title bar, click once on the spreadsheet icon with the blue field, and a pop up box will appear. Select from the list each analyte in the standard or reagent (individually). Click "OK" button. The analyte will appear in the analyte list in the bottom left of the stock page. Click on the negative symbol to collapse the tree for each analyte (if expanded). Highlight each analyte individually to activate the Aliquot field and Units drop down menu at the bottom center of the stock page. Enter the amount used in the Aliquot field and select the units of measure with the drop down menu.
3. If the standard is made on a regular basis, proceed in entering the information below first, and then perform the following sequence for borrowing the analytes from a previously made standard. The total volume units and department being entered are key to using the borrow function.
   a. In the title bar, click on the borrow icon, (one beaker replicating into two beakers). A window with a list will pop up.
   b. Scroll down the left menu and find the previously received standard that is to be borrowed.
   c. Check the box next to the stock standard to be borrowed or expand the tree (click on the "+" symbol) and the individual analytes will appear with check boxes.

d. Check the boxes of the analytes that are to be borrowed.
e. Click on the move icon in the center of the menus to transfer the analytes selected to the right menu, click "OK".
f. The analytes will appear in the analyte list on the stock page.
g. Enter the concentrations and amounts in the same manner as the individually selected analyte instructions above.
h. The analyte names and concentrations in the prepared stock solution will appear in the right lower box on the stock page.
i. When all entries are verified for correctness, click the save icon on the title bar.
j. Click on the print icon on the title bar once for each label required. All stock standards must be properly labeled.

Sample Pre-Screening

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

Reference:

SW-846 Test Methods for Evaluating Solid Wastes, Method 8000, rev.2, sec 3.2.

Scope and Applicability

Both carryover from one sample to the next and the analysis of high-concentration samples can lead to contamination of the analytical instrument. Screening of samples by an alternate method or instrumentation can eliminate costly production time losses spent cleaning and servicing instrumentation.

Important Notes

Records are to be maintained in a notebook that relates to each area of testing (i.e. volatile organics, metals, etc.). The records should include the following elements: lab assigned sample number, instrument/detector type, results or suspected background (may be a qualitative description), and corrective actions or dilutions, date, and the initials of the analyst.

Procedure

When screening samples, it is important that the analyst uses their professional judgment as to the operating conditions of the instrument being used to perform the screening, (i.e., calibrations, tune clock, QC standard analysis, etc.). Therefore, results acquired are never to be reported or formally referenced in support of the results determined through the application of the formal method cited.

Samples to be analyzed for volatiles can be screened using GC/MS (Method 8260). Samples to be analyzed for semivolatiles can be screened using GC/FID. Other screening methods are also acceptable. The analyst should use the screening results to choose an appropriate dilution factor for the analysis that will prevent system contamination yet still provide adequate sensitivity for the major constituents of the sample. Screening samples is to be used as a tool by the analyst to increase production efficiency.

Record Retention

Organize and retain all raw data in a manner that is easily accessible and archived with the completed data.

LIMS Software Revision Procedure

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

Reference

Premier Laboratory, Inc. LIMS Manual: Developed internally for use with Premier Laboratory, Inc.'s BLISS system

Premier Laboratory, Inc. Statement of Qualifications

Applicability

Personnel: Information Systems (IS) Personnel

Purpose: Making code changes for the enhancement of or corrections to the LIMS software.

Procedure

A. Enhancement or Problem identification

1. An enhancement to or the identification of a problem in the software, which constitutes the user interface to the LIMS, may be discovered by anyone in the laboratory. The issue is then formally brought to the attention of the IS personnel either verbally or submitting a Request for Work (RFW).
2. The IS department will review the issue for possible paths of resolution.
3. If necessary, the Manager of Information Systems (MIS) will meet with the affected Departmental Managers and/or Managing Members to discuss the proposed changes.
4. The code changes required to bring about the agreed upon changes will be finalized.

B. Code Changes

1. Source files containing code to be changed will be checked out of the Source Control System. Or, if necessary, new source files will be created and added to the corresponding project file.
2. The required code changes will be made. All code will be commented, internally to the code, describing the intent of the procedural portion of the code.
3. A debug version of the project(s) will be compiled and linked. The debug version will be run against the test version of the LIMS database to verify the quality and precision of the changes. If further code changes are needed to attain the required result without degradation to the existing system, they will be made and retested.

4. When the changes meet the requirements, the approval of the MIS and, if necessary, the approval of the affected Departmental Managers, a release version of the project(s) may be compiled and linked. Before the release version is generated, the version number of the project(s) will be incremented.
5. Upon the completion of the generation of the release version, it will be run against the test version of the LIMS database to verify that the release version behaves as expected.
6. All modified and new source files will be checked into the Source Control System.

C. Implementation

1. The previous versions of the affected modules will be copied to an alternate location.
2. The new release version of the affected modules will be copied to the LIMS program area of the network.
3. A notification with a description of the changes will be distributed to the Departmental Managers (may be done by e-mail).
4. When necessary, Departmental Managers and appropriate laboratory personnel will be instructed on the use of the new or changed module(s).

Data Integrity Plan

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

Reference:

2003 NELAC Standard, Chapter 5, Section 5.4.2.6 and 5.5.2.7

Distribution List

Location	Personnel
Administrative Office	Quality Manager
Test Laboratory	Supervisor

Purpose

- To describe the laboratory's Data Integrity System.
- Emphasize the paramount importance of ethics in the performance of all analytical work.
- Obtain the commitment of laboratory staff to the principle that all analyses shall be performed in a controlled and documented manner.
- To ensure that laboratory staff consistently meets the specific ethical requirements defined in this data integrity plan.

Scope

This procedure applies to all analyses performed within the laboratory's scope of accreditation.

Responsibilities

Senior managers ensure that only staff members who sign the ethics agreement are allowed to work in the laboratory.

A data integrity advisor(s) shall be appointed by senior management to inform of any need for detailed investigations. A data integrity advisor shall assure confidentiality and a receptive environment in which to privately discuss personal ethical dilemmas with staff or observed unethical practices by other members of the staff. Confidential records shall be maintained by each data integrity advisor for these discussions.

The Quality Assurance Officer shall perform in-depth review of laboratory reports and the data used to support them on a quarterly basis.

The Quality Assurance Officer shall prepare a summary of plan updates, on-going ethics/data integrity training and data integrity investigations for presentation to the Accrediting Authority Assessor(s) at the time of the on-site assessment. The summary shall cover the time period since the last on-site assessment.

Procedure

1. **Ethics Training**

   Ethics training is a required part of new employee orientation and is provided on an annual basis for all laboratory managers and staff by senior laboratory management. Initial training during orientation includes the overall organizational mission and its relationship to the absolute need for honesty and full disclosure in all analytical reporting and record-keeping. Resources where applicable ethics policy and law can be found are made available and copies are distributed.

   The initial orientation is immediately followed-up by senior laboratory management with the specifics of the laboratory's data integrity plan. Examples are described that illustrate unethical behavior and ethical behavior related to laboratory data manipulation. Laboratory standard operating procedures are reviewed with respect to proper procedure, data qualifiers, and adequacy of record keeping. Management will disclose that reports and the data generated to support them are subject to routine in-depth review.

   The organizations' response to infractions of the data integrity plan will be discussed and the trainee shall understand that infractions will be investigated in a detailed way. The consequences to an employee found to be in violation of the data integrity plan may result in immediate termination, debarment, and/or civil/criminal prosecution. Confidentiality is assured during this process.

2. **Ethics Agreement**

   Following initial ethics training and on-going annual training for laboratory managers and staff, trainees shall sign a written ethics agreement. Senior managers who provide the training shall also sign the agreement. The agreement states that the signers will not engage in any unethical practices with respect to data integrity nor will they tolerate improper behavior in others if it is observed or suspected. By signing, senior managers acknowledge their duties in upholding the spirit and intent of the data integrity system and in effectively implementing the specific requirements of the plan.

3. **Monitoring**

   Reports and the data used to support them are randomly selected by the Quality Assurance Officer (QAO) for auditing. Each calendar quarter the QAO audits 5 % or 5 data packages, which ever is more. The purpose of the review is to verify that all data integrity requirements are met. Therefore the QAO, shall have an in-depth understanding of typical inappropriate analytical behavior and be trained in the data integrity system. Refer to the SOP for data review.

Blind known reference samples may be submitted for analysis as real samples by the QAO, (blind to the analyst) as part of any project or event Data and results of the reference sample are reviewed by the QAO to verify that all data integrity requirements are met.

4. **Documentation**

Confidentiality is critical and maintained by use of locked filing cabinets and password protected electronic files. All data integrity incidents must be documented, including investigative findings and disciplinary actions. Corrective actions are recorded. If client disclosure is determined to be necessary by senior laboratory management then such disclosures and outcomes are recorded.

All data integrity documents, plans, SOPs, personal records and records of investigations shall be maintained for a period of seven years. Documents are subject to the document control system and records are subject to the records management system as described in the laboratory's quality manual and related SOPs.

Annual Review

The following Laboratory Staff have read this SOP. A copy of this completed page will be maintained in the employee training record file.

Name _____ Position _____ Date _____

Name _____ Position _____ Date _____

Name _____ Position _____ Date _____

Name _____ Position _____ Date _____

Name _____ Position _____ Date _____

Name _____ Position _____ Date _____

Name _____ Position _____ Date _____

Name _____ Position _____ Date _____

Name _____ Position _____ Date _____

Name _____ Position _____ Date _____

Name _____ Position _____ Date _____

Name _____ Position _____ Date _____

Name _____ Position _____ Date _____

Name _____ Position _____ Date _____

Peak Integration

Prepared by: _____ Laboratory Director

Reviewed and Implemented by: _____ General Manager

Reference

Test Methods for Evaluating Solid Waste, SW-846, Revision 2, December 1996, Method 8000B.

I. Applicability

Integration processes employed by analysts to integrate peak areas manually using chromatographic software during IC, GC, TOC, ICP, ICP/MS, HPLC, Flow Injection and GC/MS analyses.

II. Important Notes

It is the task of all department managers to train their analysts in the proper application of this SOP. The analyst must be consistent with peak integration technique throughout the chromatogram, and with all calibration standards and QC samples associated with the sample.

It is the responsibility of the analyst to process data in accordance with this SOP and to ensure that all integrations are made part of the permanent data record. The procedures in this SOP take primacy over any client, method, or other SOP Quality Control directive.

The analyst must consult with management if they are unsure of appropriate integration procedures.

III. Procedure for Manual Integration of Peaks

Automated peak integration software will not always integrate peaks correctly. Manual integration is used to correctly quantify peak areas that the original software integration failed to identify properly. As good laboratory practice, review all peaks to see where the integration lines are drawn.

The following are specific circumstances in which manual peak integration is justified:

- The incorrect peak is picked up by the software.
- Shift in baseline, increase or decrease in magnitude
- Baseline noise level exceeding 1:3 noise/signal ratio
- Peak splitting performed by the software
- Distinct peaks present on the shoulder of another peak
- Negative peaks present in the baseline affecting the peak

Active sites may contribute to poor peak resolution such as tailing, overlay, and splitting. In these cases the baseline must be manually drawn.

Manual integration is used only when at least one of the above conditions is present. All other cases not cited must be reviewed and approved by the Department Manager or Quality Assurance (QA) manager prior to use.

When performing manual integrations, it is best to zoom in on the baseline and use a display window of less than 0.5 minutes, when possible. Determine the average noise level of the baseline. Peak integration must start at the beginning of the peak and end on the downward tail at the average noise level of the baseline. The analyst must be consistent in determining what portion of the tail is analyte and non-analyte. Split peaks are integrated on the down side based on the size of both peaks involved in the split. Analyst experience weighs heavily in determining split peak integration. Manual integration should only be used when peaks are clearly resolved with a valley between the peaks.

Under no circumstances is peak shaving or peak enhancement allowed in order to pass a calibration or other method specific criteria. Willful use of this technique is criminal and will result in immediate termination.

Quality Assurance personnel will examine chromatographic data during internal method audits to determine compliance with the procedures in this SOP.

IV. Documentation Procedures

A. HPLC Methods

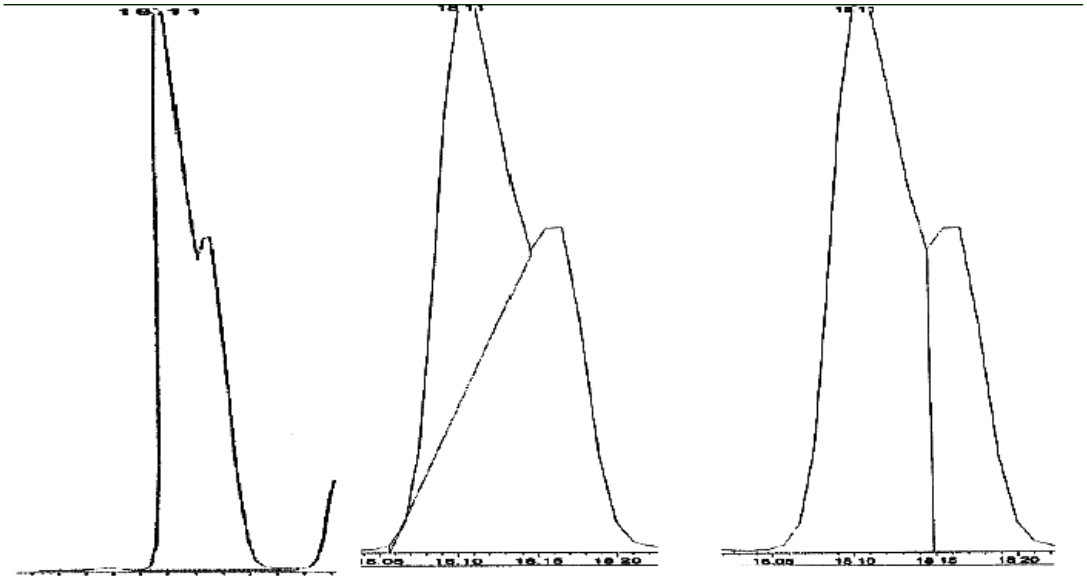
The quantitation report with the chromatogram showing the manually integrated peak(s) must be stamped "manually integrated" and initialed and dated by the analyst. Each peak that was manually integrated must be clearly identified with an "m" mark or other descriptive notation.

B. GC Methods

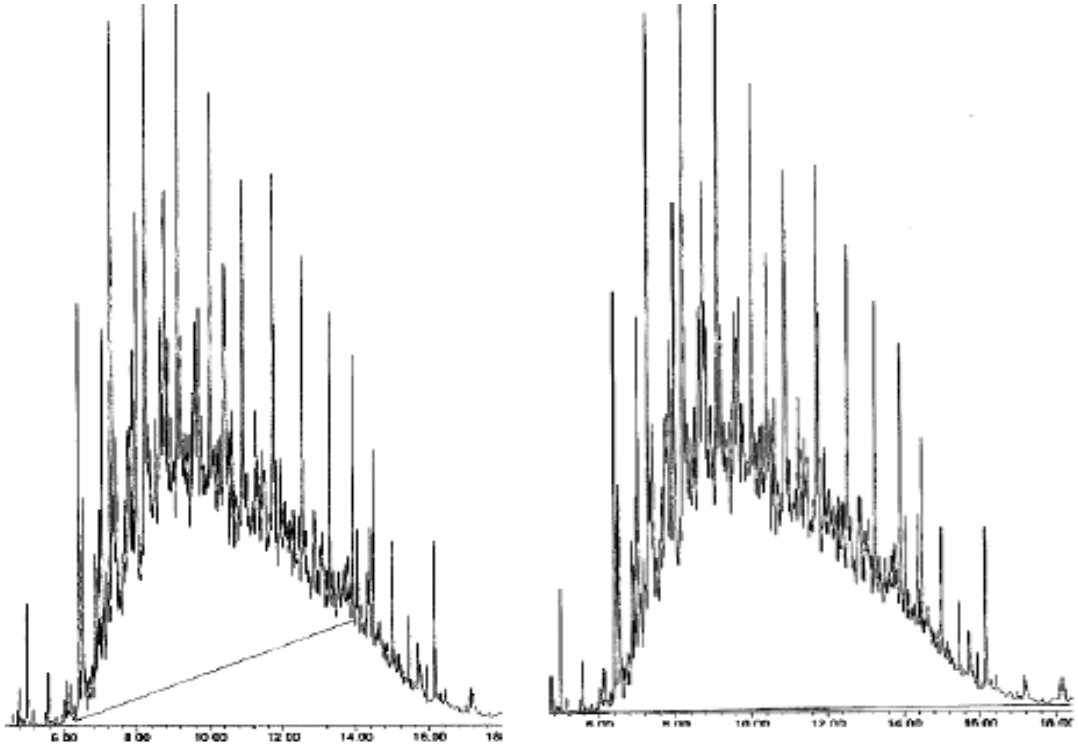
The software used on all GCs – EnviroQuant – has been programmed to identify all manually integrated peaks with an "m" on the quantitation report.

Examples of proper and improper integration:

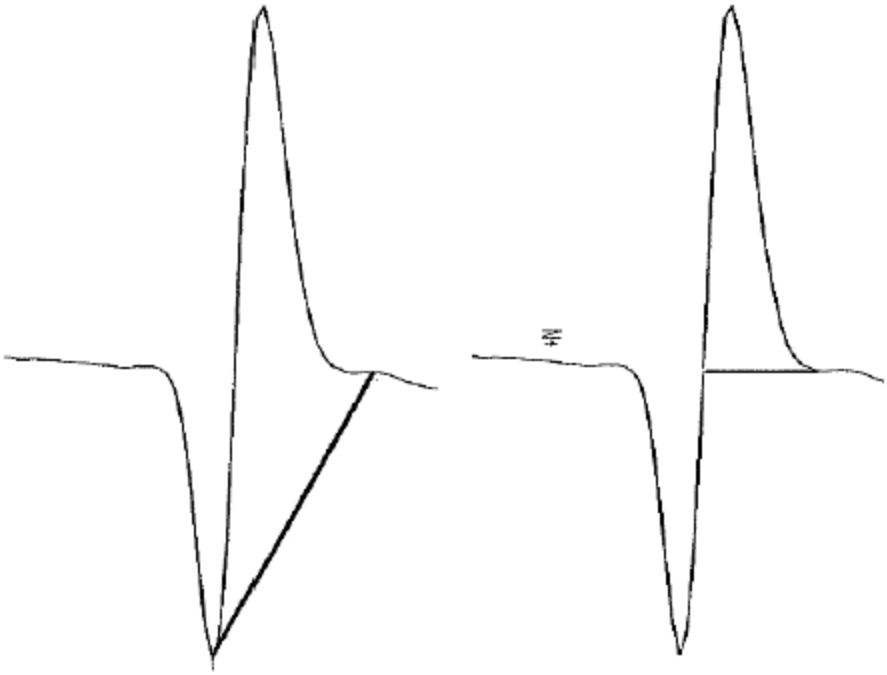
Software Integration | Improper Integration | Correct Integration



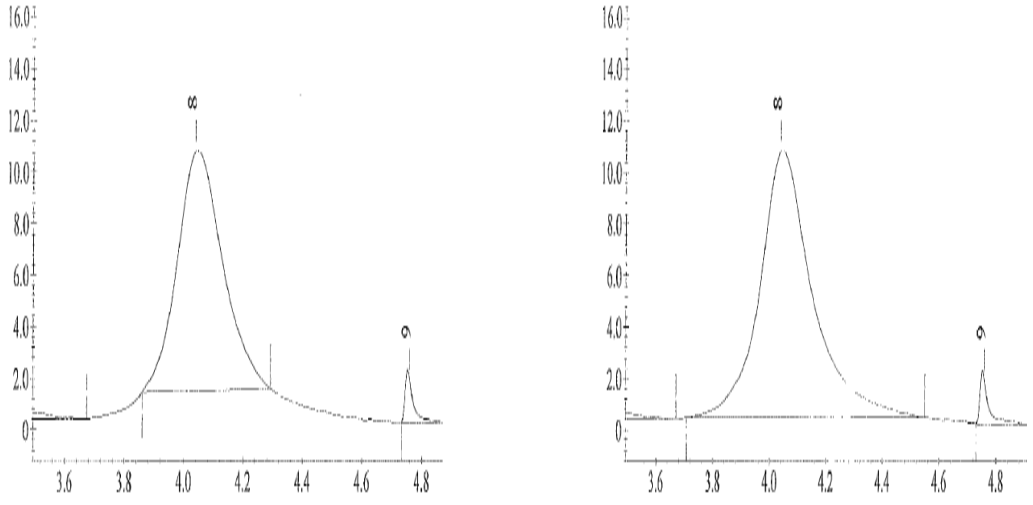
Improper Integration | Correct Integration



Improper Integration | Correct Integration



Improper Integration | Correct Integration



Sample Dilutions

Prepared by: _____ Reviewed and Implemented by: _____
Laboratory Director General Manager

Reference

Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1983

Test Methods for Evaluating Solid Waste, SW-846, 7000A, Revision 1, July 1992

Applicability

Analyte: All

Matrix: Aqueous and Solids Extracts

Regulation: NPDES, SDWA, CWA, RCRA

Important Notes

All samples submitted to the laboratory should be treated as potential health hazards.

The cleaning sequence method that must be followed for all glassware that will contact samples when making dilutions is found in the Glassware Cleaning SOP in this QM.

If filtration or similar treatment is being performed prior to dilution to eliminate physical matrix interferences the final reporting requirements must be considered. Careful attention must be paid to whether a sample is being analyzed directly or if it requires extraction, digestion or distillation prior to analysis. Always consult a Department Supervisor or a Technical Director before proceeding with a newly encountered matrix.

Procedure

Various situations arise that require a sample to be diluted prior to analysis. The following list is not exhaustive but is intended to guide decisions made at the bench level by the analyst.

Dilutions must be performed anytime:

- The sample results exceed the calibrated range for the analyte as specified in the method SOP (i.e.: bracketed range or defined percentage of highest standard).
- The sample concentrations or matrix exceed the capacity of an extract solvent, acid, oxidizer or similar used in sample preparation.
- Multiple compounds or elements are detected that may cause chromatographic separation, spectral, chemical or physical interferences.

- Matrix interference is evidenced by low spike recoveries (<30%) or suspected through observation of high levels in compounds or elements of interest.
- Duplicate analysis percent difference exceeds method requirements indicative of possible matrix interferences.

Appendix A

Math and Statistics

The following topics are included in this appendix:

A. Significant Digits
B. Rounding
C. Arithmetic Mean (Average)
D. Standard Deviation
E. Linear Regression
F. Test for Outliers

A. Significant Digits

The *n* most significant digits of a value are the left most non-zero digit and the *n-1* digits to its immediate right.

Examples: In the following numbers, the two most significant digits are underlined: 12678, 507, 8.033, 0.0003927.

B. Rounding

1. Carry at least one digit beyond the last significant digit throughout all calculations.
2. Round the final result by changing all digits beyond the last significant digit to zeroes; drop these zeroes if they are to the right of the decimal point.
   a. If the value dropped is greater than half the last significant digit, increase the last significant digit by one.

   Example: **12873 rounds to 13000**

   b. If the value dropped is less than half the last significant digit, the last significant digit remains unchanged.

   Example: **12173 rounds to 12000**

   c. If the value dropped is *exactly* half the last significant digit, the last significant digit remains unchanged if it is even (or zero) and is increased by one if it is odd.

Examples: **12500 rounds to 12000 and 13500 rounds to 14000**

C. Arithmetic Mean (Average)

$$\overline{X} = \frac{\sum X_I}{n}$$

where $\overline{X}$ = the arithmetic mean
X_I = the value of observation I
n = total number of observations

D. Standard Deviation

$$\mathbf{s} = \sqrt{\frac{\sum\left(X_I - \overline{X}\right)}{(n-1)}}$$

where: **s =** the standard deviation
X_I = the value of observation I
$\overline{X}$ = the arithmetic mean
n =total number of observations

E. Linear Regression

$$\mathbf{m} = \frac{n\sum x_I y_I - \sum x_I \sum y_I}{n\sum x_I^2 - \left(\sum x_I\right)^2}$$

$$\mathbf{b} = \frac{\sum y_I - m\sum x_I}{n}$$

$$\mathbf{r} = \frac{n\sum x_I y_I - \sum x_I \sum y_I}{\sqrt{\left(n\sum x_I^2 - \left(\sum x_I\right)^2\right)\left(n\sum y_I^2 - \left(\sum y_I\right)^2\right)}}$$

where: $\boldsymbol{x}_I$ = independent measurement
$\boldsymbol{y}_I$ = dependent measurement corresponding to $\boldsymbol{x}$,
n = total number of observations
m = the slope
b = the **y**-intercept
r = the correlation coefficient

F. Test for Outliers

The highest or lowest value in a group for which the mean and standard deviation have been calculated shall be considered an outlier if the statistic T is greater than the critical value from the table below.

$$\mathbf{T} = \frac{\overline{X} - X_I}{s}$$

where: $\overline{X}$ = the arithmetic mean for the group with $\boldsymbol{X}_I$, included
$\boldsymbol{X}_I$ = the value to be tested
s = the standard deviation for the group with $\boldsymbol{X}_I$ included

Critical Values for 1% Tests of Discordancy for a Single Outlier in a Normal Distribution			
Number of Measurements	Critical Value	Number of Measurements	Critical Value
3	1.15	15	2.71
4	1.49	16	2.75
5	1.75	18	2.82
6	1.94	20	2.88
7	2.10	30	3.10
8	2.22	40	3.24
9	2.32	50	3.34
10	2.41	60	3.41
12	2.55	100	3.60
14	2.66	120	3.66

Important Note:

All outliers found must be referenced by a narrative on the graph for the corresponding data set.

Determination of Total Organic Carbon Persulfate– Ultraviolet Oxidation Method with Nondispersive Infrared Detection (NDIR)

Prepared by: Melisa Montgomery, Quality Assurance Officer
Approved by: Robert Stevenson, Laboratory Director
Reviewed and Implemented by: Ronald Warila, General Manager

Reference

U.S. Environmental Protection Agency, Methods for the Chemical Analysis of Water and Wastes, Method 415.1 Organic Carbon, Total (Combustion or Oxidation), Approved for NPDES (Editorial Revision 1974)

U.S. Environmental Protection Agency, Methods for the Chemical Analysis of Water and Wastes, Method 415.3 Determination of Total Organic Carbon and Specific UV Absorbance at 254 nm in Source Water and Drinking Water Revision 1.0 June, 2003 (B.B. Potter, USEPA, Office of Research and Development, National Exposure Research Laboratory J.C. Wimsatt, The National Council On The Aging, Senior Environmental Employment)

U.S. Standard Methods for the Examination of Water and Wastewater, 20th edition, 5310 Total Organic Carbon (TOC), 5310C. Persulfate-Ultraviolet Oxidation method.

I. Scope and Application

1.1 This method provides procedures for the determination of total organic carbon (TOC), and dissolved organic carbon (DOC) in drinking, surface and saline waters, domestic and industrial wastes. Exclusions are noted under Definitions and Interferences.

1.2 The method is most applicable to measurement of organic carbon above 0.5 mg/L.

II. Summary

2.1 In both TOC and DOC determinations, organic carbon in the water sample is oxidized to produce carbon dioxide (CO_2), which is then measured by a detection system. The IL 500 uses the following approach for oxidizing carbon in water samples to carbon dioxide:

2.1.1 Using a UV promoted chemical oxidation with a persulfate solution. Carbon dioxide, which is released from the oxidized sample, is detected by a nondispersive infrared (NDIR) detector.

2.2 Settable solids and floating matter may cause plugging of the injection needle. To avoid this from occurring be sure that the uptake syringe is adjusted to draw from the mid-level of the 40 mL vial. The suspended matter is considered part of the sample. The resulting water sample is considered a close approximation of the original whole water sample for the purpose of TOC measurement.

2.3 The DOC procedure requires that the sample be passed through a 0.45 µm filter prior to analysis to remove particulate OC from the sample.

III. Definitions

3.1 Analysis Batch – A set of samples prepared and analyzed on the same instrument during a 24-hour period. For a TOC/DOC analysis batch, the set may contain: calibration standards, laboratory reagent blank and/or filter blanks, field blank, field samples, laboratory fortified matrix sample, field duplicate sample, and continuing calibration check standards. For a UVA analysis batch, the set may contain: filter blanks, field samples, field blank, field duplicate sample, and spectrophotometer check solutions with associated blank. An analysis batch is limited to 20 field samples. QC samples are not counted towards the 20 sample limit.

3.2 Blank – Prepared from a volume of laboratory reagent water and used as needed to fulfill quality assurance requirements and to monitor the analytical system.

3.2.1 Calibration Blank (CB) – The calibration blank is a volume of laboratory reagent water that is treated with the same reagents used in the preparation of the calibration standards. The CB is a "zero standard" and is used to calibrate the TOC instrument. The CB is made at the same time as the calibration standards and stored along with and under the same conditions as the calibration standards. The CB is also used to monitor increases in organic background found in the calibration standards over time by analyzing it as a sample and comparing the results with initial analysis of the CB.

3.2.2 Field Reagent Blank (FRB) – A volume, equivalent to that which is collected at a sample site, of laboratory reagent water is placed in a sample bottle or vial. A second empty sample bottle or vial accompanies the laboratory reagent water sample container to the sample site. At the sample site, the laboratory reagent water is transferred into the empty bottle or vial, which then becomes the FRB. The FRB is treated as a sample in all respects including shipment from the sampling site, exposure to the sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the FRB is to determine if the TOC, DOC, and UVA measurements of the samples collected in the field are free from interferences or contamination as

a result of the sample collection procedure and/or transport of the sample(s) to the laboratory. The FRB is optional and is usually used when the laboratory suspects a problem in sample collection and handling.

3.2.3 Filter Blank (FB) – The FB is an aliquot of laboratory reagent water that is filtered and analyzed using the same procedures as field samples undergoing DOC and UVA determinations. For DOC and UVA analyses, the FB serves as the LRB. The FB will give an indication of overall contribution of organic carbon contamination from laboratory sources such as the laboratory reagent water itself, labware cleaning procedures, reagents, the filter apparatus, filter, and instrument system(s).

3.2.4 Laboratory Reagent Blank (LRB) – A volume of laboratory reagent water that is prepared with each sample set and is treated exactly as a TOC sample including exposure to all glassware, plasticware, equipment, and reagents that are used with other samples. The LRB is used to determine if organic contamination or other interferences are present in the laboratory environment, reagents, apparatus, or procedures.

3.3 Calibration Solution – The following solutions are used to calibrate the TOC instrument system for TOC or DOC determinations.

3.3.1 Organic Carbon Primary Dilution Standard (OC-PDS) – A concentrated solution containing potassium hydrogen phthalate (KHP) in laboratory reagent water that is prepared in the laboratory. OC-PDS is used for the preparation of organic carbon calibration standards (OC-CAL), continuing calibration check standards (CCC) and laboratory fortified matrix samples (LFM).

3.3.2 Organic Carbon Calibration Standard (OC-CAL) – A solution prepared from the OC-PDS and diluted with LRW to various concentrations. The OC-CAL solutions are used to calibrate the instrument response with respect to organic carbon concentration.

3.3.3 Continuing Calibration Check (CCC) – An OC-CAL solution that is analyzed periodically to verify the accuracy of the existing calibration of the instrument.

3.4 Dissolved Organic Carbon (DOC) – Organic matter, contained in a water sample that is soluble and/or colloidal, that can pass through a 0.45-µm filter.

3.5 Field Duplicates (FD1 and FD2) – Two separate samples collected at the same time and placed under identical circumstances, and treated exactly the same throughout field and laboratory procedures. Analyses of FD1 and FD2 give a measure of the precision associated with sample collection, preservation, and storage, as well as laboratory procedures.

3.6 Inorganic Carbon (IC) – Carbon in water samples from non-organic sources, composed mainly from dissolved mineral carbonates and carbon dioxide. IC can interfere with the determination of TOC and DOC if it is not removed.

3.7 Laboratory Fortified Blank (LFB) – An aliquot of laboratory reagent water or other blank matrix to which a known quantity of KHP is added in the laboratory. The LFB is subjected to the same preparation and analysis as a sample. The purpose of the LFB is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. For this method, a TOC LFB is the same as a CCC and no additional LFB is required. One LFB is required with each DOC analysis batch.

3.8 Laboratory Fortified Sample Matrix (LFM) – An aliquot of a field sample to which a known quantity of KHP is added in the laboratory. The LFM is subjected to the same preparation and analysis as a sample, and its purpose is to determine whether the sample matrix affects the accuracy of the TOC or DOC analytical results. The background concentration of organic carbon in the sample matrix must be determined in a separate aliquot and the measured value in the LFM calculated.

3.9 Laboratory Reagent Water (LRW) – The laboratory reagent water is carbon free distilled water.

3.10 Material Safety Data Sheet (MSDS) – Written information provided by a vendor describing a chemical's toxicity, health hazards, physical and chemical properties (flammability, reactivity, etc.), storage, handling, and spill precautions.

3.11 Minimum Reporting Level (MRL) – The minimum concentration of organic carbon that can be reported as a quantified value in a sample following analysis.

3.12 Quality Control Sample (QCS) – A solution containing a known concentration of an organic carbon compound(s) that is analyzed exactly like a sample. The QCS is obtained from a source external to the laboratory and is different from the source used for preparing the calibration standards. It is used to check laboratory and instrument performance.

3.13 Total Carbon (TC) – A measure of the organic carbon and inorganic carbon contained in a water sample.

3.14 Total Organic Carbon (TOC) – The amount of organic carbon determined by the difference of the measured TC minus the measured IC.

IV. Interferences

4.1 Chloride *may* interfere with the persulfate oxidation method. The IL 500 uses a halide trap for chlorine interferences. The interferences will only occur, however, when the copper / brass trap is completely depleted.

4.2 Because of the possibility of oxidation or bacterial decomposition of some components of aqueous samples, the lapse of time between collection of samples and start of analysis should be kept to a minimum. Also, samples should be kept cool (4°C) and protected from sunlight and atmospheric oxygen.

4.3 In instances where analysis cannot be performed within two hours (2 hours) from time of sampling, the sample is acidified (pH < 2) with HCl or H_2SO_4.

4.4 This procedure is applicable only to homogeneous samples which can be injected into the apparatus reproducibly by means of a microliter type syringe. The opening of the syringe limits the maximum size of particles which may be included in the sample.

4.5 Sample pH check – The pH of the preserved sample or filtrate should be checked to ensure adequate acidification for the preservation. However, this should only be performed by placing a drop from the sample onto pH test paper. **Do not** put the pH paper into the sample bottle. Placing the pH paper in the sample bottle will contaminate the sample with organic carbon. If this happens, the sample or filtrate must be discarded and a new sample collected.

V. Safety

5.1 Each chemical reagent used in this method should be regarded as a potential health hazard. Exposure to these compounds should be minimized and/or avoided by active participation in safety planning and good laboratory practices. Material Safety Data Sheets (MSDS) containing information on chemical and physical hazards associated with each chemical are to be read by all personnel involved in the chemical analysis.

5.2 Potassium persulfate is a strong oxidizing and corrosive reagent. The analyst should avoid eye and skin contact by wearing eye/face protection, powder-free gloves and laboratory clothing. If body tissue comes in contact with this reagent, apply large quantities of water for at least 15 minutes (see MSDS) while removing contaminated clothing. This reagent may cause delayed burns. Seek immediate medical attention if the area becomes irritated or burned. This reagent can also cause a fire or explosion if it is allowed to come in contact with combustible materials.

5.3 Protect your hands by wearing laboratory disposable gloves during the preparation and disposal of corrosive (acids and oxidants) laboratory reagents.

VI. Equipment and Supplies

6.1 Lachat Instruments IL500 TOC automated persulfate analyzer

6.2 Filter Apparatus – hydrophilic 0.45-µm membrane filters

6.3 Injection Vials – 40 mL VOA vials, precleaned

6.4 Instrument System Software – Lachat IL 500

6.4.1 TOC Instrument – IL 500 UV/Low Temperature/Persulfate/Wet Oxidation/NDIR. The IL 500 TOC analyzer is based on UV catalyzed persulfate digestion to produce CO_2, which is then detected by an NDIR detector. The sparge is done automatically.

6.5 Laboratory Reagent Water – Ultrapure type II DI water

6.6 Muffle Furnace – A muffle furnace capable of heating up to 425°C for optional baking of non-volumetric glassware

6.7 Various Pipettes – Fixed volume pipettors

6.8 Volumetric Flasks – All volumetric glassware used in this method must be "Class A".

VII. Reagents and Standards

Important Note: ***The chemicals required for this method must be at least reagent grade.***

7.1 **Compressed Gases** – UHP grade nitrogen gas

7.2 **Laboratory Reagent Water (LRW)** – Water that has a TOC reading of <0.35 mg/L

7.3 **Disodium Hydrogen Phosphate** [Na_2HPO_4, CAS#7558-79-4] – Anhydrous, ACS grade

7.4 **o-Phosphoric Acid (85%)** [H_3PO_4, CAS#7664-38-2] – ACS grade

7.4.1 TIC Solution for IL 500 [o-Phosphoric Acid ca. 10%]: 69 ml of 85 % Phosphoric acid in 1000 ml DI water

7.5 **Sulfuric Acid** (95 - 98%) [H_2SO_4, CAS #7664-93-9] – ACS grade

7.5.1 **Acidification Solution** for standards and samples [1M H_2SO_4] – 55 mL of 95 - 98% H_2SO_4 in 1000 ml DI water

7.6 **Potassium Hydrogen Phthalate (KHP)** [$C_8H_5O_4K$, CAS#877-24-7] – Anhydrous, ACS grade

7.7 **Sodium Peroxodisulfate** [$Na_2S_2O_8$, CAS#7775-27-1] - ACS grade

7.7.1 **Reagent Solution for Wet Chemical Oxidation** – 80 g/L Sodium peroxodisulfate + 5 ml 1M H_2SO_4 per L of solution. Transfer the solution to the instrument reagent bottle.

7.8 **Standard Solutions**

7.8.1 **Inorganic Carbon Primary Test Solution (IC-TEST) Reagents** (NPOC analysis only)

7.8.1.1 **Ammonium Chloride** [NH_4Cl, CAS#12125-02-9] – ACS grade

7.8.1.2 **Calcium Chloride Dihydrate** [$CaCl_2 \cdot 2H_2O$, CAS#10035-04-8] – ACS grade

7.8.1.3 **Calcium Nitrate Tetrahydrate** [$Ca(NO_3)_2 \cdot 4H2O$, CAS#13477-34-4] – ACS grade

7.8.1.4 **Magnesium Sulfate Heptahydrate** [$MgSO_4\_7H_2O$, CAS#10034-99-8] – ACS grade

7.8.1.5 **Potassium Chloride** [KCl, CAS#7447-40-7] – ACS grade

7.8.1.6 **Sodium Bicarbonate** [$NaHCO_3$, CAS#144-55-8] – ACS grade

7.8.1.7 **Sodium Chloride** [NaCl, CAS#7647-14-5] – ACS grade

7.8.1.8 **Sodium Meta Silicate Nonahy**drate [$Na_2SiO_3 \cdot 9H_2O$, CAS#13517-24-3] – ACS grade

7.8.1.9 **Sodium Phosphate Dibasic Heptahydrate** [$Na_2HPO_4 \cdot 7H_2O$, CAS#7782- 85-6] – ACS grade

7.9 **Carbonate-Bicarbonate Stock Solution**, 1000 mg Carbon/L: Weigh 0.3500 g of sodium bicarbonate and 0.4418 g of sodium carbonate and transfer both to the same 100 mL volumetric flask. Dissolve with distilled water.

7.10 **Carbonate-Bicarbonate Standard Solution**: Prepare a series of standards at the same concentrations as the KHP for calibration of IC using the stock solution in 7.9.

7.11 **Organic Carbon Primary Dilution Standard (OC-PDS)**, 500 mg/L (1mL = 0.5 mg OC) – Prepare an acid preserved (pH <2) OC-PDS by pouring approximately 500 mL of LRW into a 1-liter volumetric flask, adding 1ml of concentrated acid for preservation, carefully transferring 1.063 g KHP into the flask, stirring until it is dissolved, and then diluting to the mark with LRW (1.0 mg KHP = 0.471 mg organic carbon). Transfer this solution to a marked amber glass reagent bottle and cap for storage. This solution does not require refrigeration for storage and is stable for an indefinite period of time (6 months to a year). Replace the OC-PDS if the instrument system fails to pass QCS requirements from a weekly made QCS.

VIII. Procedure

8.1 Instrument Set Up and Optimization

8.1.1 Prior to calibrating the TOC instrument IL 500, clean the instrument system with carbon dioxide free water and sparge reagents with ultra high purity reagent gas as specified by the Instrument Manual to remove background carbon dioxide.

8.1.2 Monitor the instrument background carbon dioxide levels for at least 5-15 minutes. The IL 500 instrument should have a stable background and be free from drift caused by CO_2 contaminated gas or leaks in the system.

8.1.3 After the instrument is judged to be stable, load the auto-injector and inject four LRB samples and start the analysis. The data collected from the first injection of LRB is discarded and is considered a system cleanup blank. The next three LRB injections should produce consecutive readings that fall within 20% of their mean.

8.1.4 If these conditions are met, the instrument is ready for calibration. If not, use the working standard E and repeat this section. If the three injections of working standard E do not produce consecutive readings that fall within 20% of their mean, the instrument is not ready to operate and maintenance must be performed according to the instrument operation manual before proceeding.

8.2 Autosampler Setup

8.2.1 Place the standards in the autosampler in order of increasing concentration and perform analysis. Place in order, LRB, low-level CC, LFM, followed by the first 10 or fewer samples, CCC (varied conc.), last 10 or fewer samples, and closing CCC.

8.2.2 In summary, at least one low CCC and one mid-CCC are analyzed with each analysis batch in order to verify the calibration curve.

8.2.3 Low, mid, and high CCCs are to be used to verify the calibration curve over time.

8.3 Calibration

8.3.1 For calibration of the instrument, prepare a curve with a series of 4 standards and a read blank encompassing the reporting range of the samples. The correlation coefficient must be 0.995 or better for the calibration to be considered valid over the calibration range.

8.3.2 Filtration of the CAL standards for DOC analysis is unnecessary, since interferences from the filtration unit are monitored via the FB.

8.3.3 After the calibration has been established, it must be verified by the analysis of a suitable quality control sample (QCS). If measurements exceed +/- 10% of the established QCS value, the analysis should be terminated and the instrument recalibrated.

8.3.4 The calibration must be verified prior to beginning the each analysis batch (24 hour clock) by running a continuing calibration check. If measurements exceed ± 20% of the established mid level CC value, the analysis should be terminated and the instrument recalibration must be performed.

8.3.5 Periodic reanalysis of the QCS is to be performed as an additional continuing calibration check.

8.4 TOC Sample

8.4.1 The TOC sample is collected in two 40-mL injection vials, each acidified to pH <2 by adding 2 drops of concentrated acid. TOC samples must be acidified at the time of collection. Cap each injection vial and invert several times to mix the acid.

8.4.2 Samples shipped that are improperly preserved, and/or do not arrive at the laboratory within 48 hrs, cannot be used for compliance monitoring under the SDWA.

8.4.3 The sample is stored at 4- 6 °C, until analysis. Stored and preserved samples must be analyzed within 28 days from time of collection.

8.5 The DOC sample must be filtered in the field or in the laboratory through a 0.45 µm membrane filter within 48 hours of sample collection prior to acidification and analysis. After filtration, the DOC sample is acidified with concentrated acid drop wise to a pH <2. The DOC bottle is capped and inverted several times to mix the acid and is stored at 4-6 °C. The sample must be analyzed within 28 days from time of collection.

IX. Data Analysis and Calculations

9.1 Sample concentrations are computed by the instrument software by subtracting the measured IC from the measured TC. Multiply answers by any applicable dilution factor performed during analysis.

9.2 Report only those values that fall between the lowest and the highest calibration standards. Samples exceeding the highest standard should be diluted and reanalyzed.

9.3 Results are reported in mg/L TOC.

X. Quality Control

10.1 The minimum requirements for this method consists of an initial demonstration of laboratory capability, and the periodic analysis of laboratory reagent blanks, fortified blanks and other laboratory solutions as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of the data that are generated.

10.2 Initial Demonstration of Performance

10.2.1 The initial demonstration of performance is used to characterize instrument performance (determination of LCRs and analysis of QCS) and laboratory

performance (determination of MDLs) prior to performing analyses by this method.

10.2.2 **Initial Demonstration of Accuracy** – The initial demonstration of accuracy consists of the analysis of five (5) LFBs analyzed as samples at a concentration between 2 to 5 mg/L OC. If DOC analysis is being performed, the LFB must be filtered through a 0.45um membrane filter. The average recovery between 2 to 5 mg/L OC must be within ± 20% of the true value. If ± 20%of the true value is exceeded, identify and correct the problem and repeat.

10.2.3 **Initial Demonstration of Precision** – Calculate the average precision of the replicates in the Initial Demonstration of Accuracy. The RSD% must be no greater than 20%. If the RSD% exceeds 20%, identify and correct the problem and repeat Sections 10.1.2.

10.2.4 **Quality Control Sample (QCS)** – When beginning the use of this method, on a quarterly basis or as required during recalibration, verify the calibration standards and acceptable instrument performance with the preparation and analyses of a QCS. If the determined concentrations are not within 20% of the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before either proceeding with the initial determination of MDLs or continuing with on-going analyses.

10.2.5 **Method Detection Limit (MDL)** – MDLs must be established using reagent water (blank) fortified at a concentration of two to three times the estimated instrument detection limit.

10.2.5.1 To determine MDL values, take seven replicate aliquots of the fortified reagent water and process through the entire analytical method. Perform all calculations defined in the method and report the concentration values in the appropriate units. Calculate the MDL as follows:

$$\mathbf{MDL = (t) \times (S)}$$

where: t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom [t= 3.14 for seven replicates]

S = standard deviation of the replicate analyses

10.2.5.2 MDLs must be determined annually, when a new operator begins work or whenever there is a significant change in the background or instrument response.

10.3 Assessing Laboratory Performance:

10.3.1 **Laboratory Reagent Blank (LRB)** – The laboratory must analyze at least one LRB with each batch of 20 samples or less. Data produced is used to assess contamination from the laboratory environment. Values that exceed the MDL indicate laboratory or reagent contamination should be suspected and corrective actions must be taken before continuing the analysis.

10.3.2 **Laboratory Fortified Blank (LFB)** – At least one LFB with each batch of 20 samples or less. In TOC analysis there is no difference in the composition of the LFB compared to the CC, therefore the CC may be evaluated as the LFB. This is not true with DOC, and a separate LFB processed through filtration must be evaluated. Calculate accuracy as percent recovery (Section 10.4.2). If the recovery of any analyte falls outside the required control limits of 80-120%, that analyte is judged out of control, and the source of the problem should be identified and resolved before continuing analyses.

10.3.3 The LFB analyses data must be used to assess laboratory performance against the required control limits of 80-120%. When sufficient internal performance data become available (usually a minimum of 20-30 analyses), optional control limits can be developed from the percent mean recovery (x) and the standard deviation (S) of the mean recovery. These data can be used to establish the upper and lower control limits as follows:

Upper Control Limit = x + 3S
Lower Control Limit = x - 3S

The optional control limits must be equal to or better than the required control limits of 90-110%. After each five to 10 new recovery measurements, new control limits can be calculated using only the most recent 20-30 data points. Also, the standard deviation (S) data should be used to establish an on-going precision statement for the level of concentrations included in the LFB.

10.3.4 **Continuing Calibration Check Solution (CCC)** – A low level CCC solution with a concentration at the method reporting limit must be analyzed prior to starting a run sequence on a 24 hour clock and be within 50% of the true value. Subsequent analysis of either a mid-level or high-level CCC solution must be run after the first 10 samples and after the last sample. The recovery of the mid-level CCC must be within 20% of the true value and the analysis of a high- level CC solution must be within 15% of the true value. If the calibration cannot be verified within the specified limits, reanalyze the CCC solution. If the second analysis of any CCC solution confirms the calibration to be outside the limits, sample analysis

must be discontinued, the cause determined and/or in the case of drift, the instrument recalibrated. All samples following the last acceptable CCC solution must be reanalyzed. The analysis data of the calibration blank and CCC solution must be kept on file with the sample analyses data.

10.4 Assessing Analyte Recovery and Data Quality:

10.4.1 **Laboratory Fortified Sample Matrix (LFM)** – Within each TOC or DOC analysis batch, an aliquot of one field sample is fortified at the same level as the LFB spike. In each case the LFM aliquot must be a duplicate of the aliquot used for sample analysis.

10.4.1.1 If the concentration of fortification is less than 25% of the background concentration of the matrix the matrix recovery should not be calculated.

10.4.1.2 Calculate the percent recovery for each analyte, corrected for concentrations measured in the unfortified sample, and compare these values to the designated LFM recovery range 75-125%. Percent recovery may be calculated using the following equation:

$$\mathbf{R = \frac{C_s - C \times 100}{S}}$$

where: R = percent recovery
C = fortified sample concentration
C_s = sample background concentration
s = concentration equivalent of analyte added to sample

10.4.1.3 Until sufficient data becomes available (usually a minimum of 20-30 analysis), assess laboratory performance against recovery limits of 75-125%. When sufficient internal performance data becomes available, develop control limits from percent mean recovery and the standard deviation of the mean recovery.

10.4.1.4 If the recovery of any analyte falls outside the designated LFM recovery range and the laboratory performance for that analyte is shown to be in control (LFB), the recovery problem encountered with the LFM is judged to be either matrix or solution related, not system related.

10.4.2 **Sample Duplicate** – Analyze one duplicate sample for every 20 samples. A duplicate sample is a sample brought through the entire sample preparation and analytical process. A control limit of ± 20% for RPD shall be used for sample values greater than 5 times the instrument detection limit. A difference of detection limit is to be used to evaluate samples below 5 times the detection limit.

XI. Pollution Prevention

11.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice.

11.2 Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

11.3 Quantity of the chemicals purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

SOP Revision History

Date	Previous Revision No.	New Revision No.	Description of Changes	Effective Date	Initiated by
7/27/11	1.0	1.1	Added revision history table and changed format	7/27/11	LM

ICP Metals
Method 6010B

Prepared by: Melisa Montgomery, Quality Assurance Officer
Approved by: Robert Stevenson, Laboratory Director
Reviewed and Implemented by: Ronald Warila, General Manager

Reference

Test Methods for Evaluating Solid Waste, SW-846, Revision 2, December 1996, Method 6010B

I. Applicability

1.1 Analyte: Refer to ICP manual for installed spectral lines

1.2 Matrix: Digestates from procedures 3005A, 3010A, 3015, 3040A, 3051

1.3 Regulation: RCRA

II. Important Notes

2.1 The proper identification of interferences encountered while performing ICP analysis is vital to producing sound analytical data. The following is a brief summary of some of the major interferences that may produce either false positive or false negative results.

2.2 Spectral interferences are caused by (1) overlap of a spectral line from another element; (2) unresolved overlap of molecular band spectra; (3) background contribution from continuous or recombination phenomenon and (4) stray light from the line emission of high-concentration elements. Computer-correcting the raw data after monitoring and measuring the interfering element can compensate for spectral overlap. Unresolved overlap requires selection of an alternate wavelength. Background contribution and stray light can usually be compensated for by a background correction adjacent to the analyte line.

2.3 Users of simultaneous multi-element instruments must verify the absence of spectral interference from an element in a sample for which there is no instrument detection channel. Potential spectral interferences for the recommended wavelengths are given in Table 2. The data in Table 2 are intended as rudimentary guides for indicating potential

interferences; for this purpose, linear relations between concentration and intensity for the analytes and the interference can be assumed.

2.4 The interference is expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that As is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Al. According to Table 2, 100 mg/L of Al would yield a false signal for As equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of Al would result in a false signal for As equivalent to approximately 0.13 mg/L. The interference effects must be evaluated for each individual instrument since the intensities will vary with operating conditions, power, viewing height, argon flow rate, etc.

2.5 Generally, interferences were discernible if they produced peaks, or background shifts, corresponding to 2 to 5% of the peaks generated by the analyte concentrations.

2.6 At present, information on the listed silver and potassium wavelengths is not available, but it has been reported that second-order energy from the magnesium 383.231-nm wavelength interferes with the listed potassium line at 766.491 nm.

2.7 Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample, by using a peristaltic pump or by using the standard additions method. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, which affects aerosol flow rate and causes instrumental drift. The problem can be controlled by wetting the argon prior to nebulization, using a tip washer, or diluting the sample. Also, it has been reported that better control of the argon flow rate improves instrument performance; this is accomplished with the use of mass flow controllers.

2.8 Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique. If observed, they can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by buffering the sample, by matrix-matching and by standard addition procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element.

III. Procedure

3.1 Preliminary treatment of most matrices is necessary due to the complexity of sample matrices. The use of an internal standard or matrix matching must be used to determine concentrations of unknowns. The internal standard used is yttrium.

3.2 Set up the instrument with proper operating parameters established by the instrument manufacturer. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 30 minutes of operation prior to calibration).

3.3 Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions described in Table 3. Flush the system with a reagent blank between each standard. Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error. In the case of multi-level calibrations the correlation coefficient must be > 0.995 for all elements.

3.3.1 **NOTE:** For boron concentrations greater than 500 mg/L, extended flush times >1 minute may be required.

3.4 The validity of the calibration must be verified by analyzing a second source standard (ICV) with concentrations of all elements of interest at or near the midpoint of the calibration.

3.5 The CCV and ICB must be run and meet the QC requirements before proceeding. Each may be rerun once before having to initiate corrective actions and recalibration.

3.6 Flush the system with the calibration blank solution for at least 1 minute before the analysis of each sample. Rinse time may be reduced if data will support the absence of analytes above the stated MDLs.

3.6.1 Analyze the instrument performance check and the calibration blank after every 10 samples.

3.7 An Interference Check solution (ICS) containing the major interferences must be run prior to analysis of samples.

3.7.1 An ICSA and ICSAB must also be run at the beginning and end of each analytical sequence, this solution contains parts A (major interferences) only and A (major interferences) plus B (elements of interest), respectively.

3.8 A low-level check standard (or project required detection limit standard, however named) must be run at the beginning of each run and at the end.

3.8.1 Unsatisfactory recoveries must be narrated in the final report based on project specific limits and requirements.

IV. Standards Preparation

4.1 Standard Concentrations: Enviro 61E

4.1.1 Standard 1 (serves as the CCB/ICB) – add 10 mL HNO_3 to 490 mL reagent water.

Standard 2 – This standard serves as the initial calibration standard for the analytes below					
Stock Solution	**Concentration, ppm**	**Amount, mL**	**Stock Standard**	**Concentration, ppm**	**Amount, mL**
Ag	1000	2.5	Ni	1000	5.0
As	1000	5.0	Pb	1000	5.0
B	1000	5.0	Sb	1000	5.0
Ba	10,000	2.0	Se	1000	5.0
Be	1000	1.0	Si	1000	5.0
Cd	1000	5.0	Sn	1000	5.0
Co	1000	5.0	Ti	1000	5.0
Cr	1000	2.5	Tl	1000	5.0
Cu	1000	2.5	V	1000	5.0
Mn	1000	5.0	Zn	1000	5.0
Mo	1000	5.0			
Volume HNO_3, mL = 10				**Volume reagent H_2O, mL = 399.5**	
Total Vol., mL = 500					

Standard 3 – This standard serves as the initial calibration standard for the analytes below		
Stock Solution	**Concentration, ppm**	**Amount, mL**
Al	10,000	2.5
Ca	10,000	5.0
Fe	10,000	1.0
Mg	10,000	5.0
K	10,000	5.0
Na	10,000	5.0

Vol. HNO_3, mL = 10	**Vol. reagent H_2O, mL = 466.5**
Total Vol., mL = 500	

ICV			
Stock Solution	**Concentration, ppm**	**Element(s)**	**Amount, mL**
*CLPP-CAL 1	5000	Ca, Mg, K, Na	20
	1000	Al, Ba, Fe	
	500	Co, Mn, Ni, V, Zn	
	250	Ag, Cu	
	200	Cr	
	50	Be	
*CLPP-CAL 2	1000	Sb	10
*CLPP-CAL 3	1000	As, Pb, Se, Tl	10
	50	Cd	
Mo Standard Solution	1000	Mo	10
Ti Standard Solution	1000	Ti	10
Vol. HNO_3, mL = 40		**Vol. reagent H_2O, mL = 1920**	
Total Vol., mL = 2000			
***(Certified Vendor)**			

CCV
The CCV solution must be made from the same stock solutions as the calibration standards.
CCV solutions can be made using the corresponding 61E Standards at a dilution of 1:5. In the instances where instability is not an issue, a combination of 61E Standards is acceptable.

ICSAB			
Stock Solution	**Concentration, ppm**	**Element(s)**	**Amount, mL**
**CLPP-ICS-A	5000	Al, Ca, Mg	100
	2000	Fe	
**CLPP-ICS-B	100	Cd, Pb, Ni, Ag, Zn	10
	50	Ba, Be, Cr, Co, Mn, V	
As	1000		2.0
B	1000		1.0
Mo	1000		1.0
Sb	1000		1.0
Se	1000		1.0
Si	1000		1.0
Sn	1000		1.0
Ti	1000		1.0
Vol. HNO_3, mL = 20		**Vol. reagent H_2O, mL = 861**	
Total Vol., mL = 1000			

ICSA			
Stock Solution	**Concentration, ppm**	**Element(s)**	**Amount, mL**
**CLPP-ICS-A	5000	Al, Ca, Mg	100
	2000	Fe	
**CLPP-ICS-A	100	Cd, Pb, Ni, Ag, Zn	10
	50	Ba, Be, Cr, Co, Mn, V	
Vol. HNO_3, mL = 100		**Vol. reagent H_2O, mL = 790**	
Total Vol., mL = 1000			

**(Certified Vendor)

Yttrium Internal Standard		
Stock Solution		
***Yttrium Solid, g**	**Vol. HNO_3, mL**	**Vol. Reagent H_2O, mL**
0.39	2	98
Yttrium nitrate tetrahydrate, 99.999%, or Yttrium Oxide, 99.9999% The use of yttrium oxide requires the solution be gently heated to dissolve.		

Working Solution			
Vol. Stock Solution, mL	**Vol. HNO_3, mL**	**Vol. Reagent H_2O, mL**	**Total Vol., mL**
50	20	930	1000
This solution is not used to determine a specific concentration but a constant absorbance, and therefore has no QC required recovery limits.			

61E Profile Solution			
***Vol. Cu Stock, mL**	**Vol. HNO_3, mL**	**Vol. Reagent H_2O, mL**	**Total Vol., mL**
1.0	**2.0**	**97**	**100**
This solution is used to generate a peak on the copper line in order to profile the instrument; the final solution is ≈ 10ppm.			

Individual Element Concentrations in Standards (ppb)								
Element	**Standard**							
	STD1	**STD2**	**STD3**	**CCV**	**ICV**	**ICSA**	**ICSAB**	**LFB**
Ag	Blank	5,000	-	1,000	2,500	-	1,000	500
Al		-	50,000	10,000	20,000	250,000	250,000	10,500
As		10,000	-	2,000	5,000	-	2,000	500
B		10,000	-	2,000	5,000	-	1,000	500
Ba		40,000	-	8,000	20,000	-	500	500
Be		2,000	-	400	500	-	500	500
Ca		-	100,000	20,000	50,000	250,000	250,000	10,500
Cd		10,000	-	2,000	2,500	-	1,000	500
Co		10,000	-	2,000	5,000	-	500	500
Cr		5,000	-	1,000	2,000	-	500	500
Cu		5,000	-	1,000	2,500	-	500	500
Fe		-	20,000	4,000	10,000	100,000	100,000	500
K		-	100,000	20,000	50,000	-	-	25,000
Mg		-	100,000	20,000	50,000	250,000	250,000	10,500
Mn		10,000	-	2,000	5,000	-	500	500
Mo		10,000	-	2,000	5,000	-	1,000	500
Na		-	100,000	20,000	50,000	-	-	10,500
Ni		10,000	-	2,000	5,000	-	1,000	500
Pb		10,000	-	2,000	5,000	-	1,000	500
Sb		10,000	-	2,000	5,000	-	1,000	500
Se		10,000	-	2,000	5,000	-	1,000	500
Sn		10,000	-	2,000	5,000	-	1,000	500
Ti		10,000	-	2,000	5,000	-	1,000	500
Tl		10,000	-	2,000	5,000	-	1,000	500
V		10,000	-	2,000	5,000	-	500	500
Zn		10,000	-	2,000	5,000	-	1,000	500

Multipoint calibration is performed using the listed standards as is, @1:2 dilutions, and @1:4 dilutions.

A low level standard is run for all elements of interest at or near specific project reporting limits when applicable.

4.2 Standard Concentrations and Preparation - Trace

Trace 1 – This standard serves as the CCB/IC		
Vol. HNO_3, mL	**Vol. Reagent H_2O, mL**	**Total Vol., mL**
10	490	500

Trace 2 – This standard serves as the initial calibration standard for the analytes below					
Stock Solution	**Concentration, ppm**	**Amount, mL**	**Stock Standard**	**Concentration, ppm**	**Amount, mL**
Ag	1000	0.5	Mn	1000	0.5
As	1000	0.5	Mo	1000	0.5
B	1000	2.5	Ni	1000	0.5
Ba	10,000	0.25	Pb	1000	0.5
Be	1000	0.5	Sb	1000	2.5
Cd	1000	0.5	Se	1000	2.5
Co	1000	0.5	Ti	1000	0.5.
Cr	1000	0.5	Tl	1000	0.5
Cu	1000	0.5	V	1000	0.5
Volume HNO_3, mL = 10			**Volume reagent H_2O, mL = 475.25**		
Total Vol., mL = 500					

Trace 3 – This standard serves as the initial calibration standard for the analytes below.		
Stock Solution	**Concentration, ppm**	**Amount, mL**
Al	10,000	0.5
Ca	10,000	0.5
Fe	10,000	1.0
Mg	10,000	0.5
Na	10,000	5.0
Vol. HNO_3, mL = 10	**Vol. reagent H_2O, mL = 482.5**	
Total Vol., mL = 500		

Trace 4 – This standard serves as the initial calibration standard for the analytes below.		
Stock Solution	**Concentration, ppm**	**Amount, mL**
Sn	1000	2.5
Zn	1000	0.5
Vol. HNO_3, mL = 10	**Vol. reagent H_2O, mL = 487**	
Total Vol., mL = 500		

Trace 5 – This standard serves as the initial calibration standard for the analyte below.		
Stock Solution	**Concentration, ppm**	**Amount, mL**
K	10000	2.5
Vol. HNO_3, mL = 10	**Vol. reagent H_2O, mL = 487.5**	
Total Vol., mL = 500		

ICV

This sample serves as the quality control sample for the analytes below.

All stock solutions used must be from a source independent of the calibration standards.

Stock Solution	Concentration, ppm	Amount, mL	Stock Standard	Concentration, ppm	Amount, mL
61E ICV	Varied	100	Fe	10,000	0.9
Ag	1000	0.25	K	10,000	2.0
B	1000	2.0	Na	10,000	0.45
Be	1000	0.5	Sb	1000	2.0
Cd	1000	0.25	Se	1000	2.0
Cr	1000	0.25	Sn	1000	2.0
Cu	1000	0.25			

Volume HNO_3, mL = 20 **Volume reagent H_2O, mL = 867.15**

Total Vol., mL = 1000

CCV

The CCV solution must be made from the same stock solutions as the calibration standards.

CCV solutions can be made using the corresponding Trace Standards at a dilution of 1:5. In the instances where instability is not an issue, a combination of Trace Standards is acceptable.

ICSAB			
Stock Solution	**Concentration, ppm**	**Element(s)**	**Amount, mL**
**CLPP-ICS-A	5000	Al, Ca, Mg	25
	2000	Fe	
**CLPP-ICS-B	100	Cd, Pb, Ni, Ag, Zn	2.5
	50	Ba, Be, Cr, Co, Mn, V	
As	1000		0.5
B	1000		0.25
Mo	1000		0.25
Sb	1000		0.25
Se	1000		0.25
Sn	1000		0.25
Ti	1000		0.25
Vol. HNO_3, mL = 20		**Vol. reagent H_2O, mL = 950.5**	
Total Vol., mL = 1000			
****(Certified Vendor)**			

Alternately, the TRACE SICAB may be created through a 1:4 dilution of the 61E SICAB.

ICSA- The ICSA is created through a 1:4 dilution of the 61E ICSA solution.

Trace Only – Yttrium/Lithium Internal Standard		
Stock Solutions		
***Yttrium Solid, g**	**Vol. HNO_3, mL**	**Vol. Reagent H_2O, mL**
0.39	2	98
Lithium Solid, g	**Vol. HNO_3, mL**	**Vol. Reagent H_2O, mL**
0.25	2	98
*Yttrium nitrate tetrahydrate, 99.999%, or Yttrium Oxide, 99.9999% The use of yttrium oxide requires the solution be gently heated to dissolve.		

Working Solution				
Vol. Yttrium Stock Solution, mL	**Vol. Lithium Stock Solution, mL**	**Vol. HNO_3, mL**	**Vol. Reagent H_2O, mL**	**Total Vol., mL**
25	5.0	20	950	1000
This solution is not used to determine a specific concentration but a constant absorbance, and therefore has no QC required recovery limits.				

Trace Profile Solution			
***Vol. 1000 ppm As Stock, mL**	**Vol. HNO_3, mL**	**Vol. Reagent H_2O, mL**	**Total Vol., mL**
0.5	**2.0**	**97.5**	**100**
In trace analysis, this solution is used to generate a peak on the copper line in order to profile the instrument; the final solution is ≈ 5 ppm.			

Individual Element Concentrations in Standards (ppb)										
Element	**Standard**									
	Trace 1	**Trace 2**	**Trace 3**	**Trace 4**	**Trace 5**	**CCV**	**ICV**	**ICSA**	**ICSAB**	**LFB**
Ag	Blank	1,000	---	---	---	500	500	---	250	500
Al		---	10,000	---	---	5,000	2,000	62,500	62,500	10,500
As		1,000	---	---	---	500	500	---	500	500
B		5,000	---	---	---	2,500	2,000	---	250	500
Ba		5,000	---	---	---	2,500	2,000	---	125	500
Be		1,000	---	---	---	500	550	---	125	500
Ca		---	10,000	---	---	5,000	5,000	62,500	62,500	10,500
Cd		1,000	---	---	---	500	500	---	250	500
Co		1,000	---	---	---	500	500	---	125	500
Cr		1,000	---	---	---	500	450	---	125	500
Cu		1,000	---	---	---	500	500	---	125	500
Fe		---	20,000	---	---	10,000	10,000	25,000	2,500	2,500
K		---	---	---	50,000	25,000	25,000	---	---	25,000
Mg		---	10,000	---	---	5,000	5,000	62,500	62,500	10,500
Mn		1,000	---	---	---	500	500	---	125	500
Mo		1,000	---	---	---	500	500	---	250	500
Na		---	100,000	---	---	50,000	50,000	---	---	10,500
Ni		1,000	---	---	---	500	500	---	250	500
Pb		1,000	---	---	---	500	500	---	250	500
Sb		5,000	---	---	---	2,500	2,500	---	250	500
Se		5,000	---	---	---	2,500	2,500	---	250	500
Sn		---	---	5,000	---	2,500	2,500	---	250	500
Ti		1,000	---	---	---	500	500	---	250	500
Tl		1,000	---	---	---	500	500	---	250	500
V		1,000	---	---	---	500	500	---	125	500
Zn		---	---	1,000	---	500	500	---	250	500

Multipoint calibration is performed using the listed standards as is, @1:2 dilutions, and @1:4 dilutions with the exception of Trace 3, which is diluted 1:10 instead of 1:4. All standard dilutions must be properly recorded in the working standards logbook.

A low-level standard is run for all elements of interest at or near specific project reporting limits when applicable.

Potential Interferences

Analyte Concentration Equivalents Arising from Interferences at the 100 mg/L Level[a]

Analyte	Wavelength, nm	Interferent[a,b]									
		Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Tl	V
Aluminum	308.215	--	--	--	--	--	--	0.21	--	--	1.4
Antimony	206.833	0.47	--	2.9	--	0.08	--	--	--	0.25	0.45
Arsenic	193.696	1.3	--	0.44	--	--	--	--	--	--	1.1
Barium	455.403	--	--	--	--	--	--	--	--	--	--
Beryllium	313.042	--	--	--	--	--	--	--	--	0.04	0.05
Cadmium	226.502	--	--	--	--	0.03	--	--	0.02	--	--
Calcium	317.933	--	--	0.08	--	0.01	0.01	0.04	--	0.03	0.03
Chromium	267.716	--	--	--	--	0.003	--	0.04	--	--	0.04
Cobalt	228.616	--	--	0.03	--	0.005	--	--	0.03	0.15	--
Copper	324.754	--	--	--	--	0.003	--	--	--	0.05	0.02
Iron	259.940	--	--	--	--	--	--	0.12	--	--	--
Lead	220.353	0.17	--	--	--	--	--	--	--	--	--
Magnesium	279.079	--	0.02	0.11	--	0.13	--	0.25	--	0.07	0.12
Manganese	257.610	0.005	--	0.01	--	0.002	0.002	--	--	--	--
Molybdenum	202.030	0.05	--	--	--	0.03	--	--	--	--	--
Nickel	231.604	--	--	--	--	--	--	--	--	--	--
Selenium	196.026	0.23	--	--	--	0.09	--	--	--	--	--
Sodium	588.995	--	--	--	--	--	--	--	--	0.08	--
Thallium	190.864	0.30	--	--	--	--	--	--	--	--	--
Vanadium	292.402	--	--	0.05	--	0.005	--	--	--	0.02	--
Zinc	213.856	--	--	--	0.14	--	--	--	0.29	--	--

[a] Dashes indicate that no interference was observed even when interferents were introduced at the following levels:

Al -	1000 mg/L	Mg -	1000 mg/L
Ca -	1000 mg/L	Mn -	200 mg/L
Cr -	200 mg/L	Tl -	200 gm/L
Cu -	200 mg/L	V -	200 mg/L
Fe -	1000 mg/L		

[b] The figures recorded as analyte concentrations are not the actual observed concentrations; to obtain those figures, add the listed concentrations to the interferent figure.

[c] Interferences will be affected by background choice and other interferences that may be present.

Additional interference corrections are required with an axial view instrument.

V. Quality Assurance

5.1 All quality control data should be maintained and available for easy reference or inspection.

5.2 Calibration Solutions

5.2.1 The calibration solutions are made using the same or similar acid matrix as the samples to be analyzed.

5.3 High Standards Check (HSC)

5.3.1 The HSC is the highest level standard applied in a multi-point calibration for each analyte of interest. The HSC is run immediately after the calibration when required to meet specific project requirements. The HSC recovery must be within ± 5% of the true value for each analyte of interest.

5.4 Initial Calibration Verification (ICV)

5.4.1 The ICV must be made from an outside second source different from that of the calibration standards' stock solutions.

5.4.2 The ICV is used to verify initially the calibration standards or stock solutions. The ICV must be run following the calibration. The ICV recovery must be within ±10% of the true value for each analyte of interest.

5.5 Continuing Calibration Verification (CCV)

5.5.1 The CCV must be run periodically (every 10 samples) and at the end of each analytical sequence. The CCV is made from the same source as the calibration standards.

5.5.2 All recoveries must be ±10% of the true value. The CCV may be run one additional time if the specified recoveries are not met, however if the second analysis fails, corrective action must be taken and any samples analyzed after the previous valid CCV must be re-analyzed.

5.6 Calibration Blank

5.6.1 The calibration blank contains the same acid matrix as the calibration standards and run with the ICV. The calibration blank is also used as the Continuing Calibration Blank (CCB) solution. See note 1.

5.6.2 The results of the calibration blank are to agree within two standard deviations of the mean blank value. If not, repeat the analysis two more times and average the results.

5.6.3 If the average is not within three standard deviations of the background mean, terminate the analysis, correct the problem; re-calibrate, and reanalyze the previous 10 samples.

5.7 Laboratory Reagent Blank (LRB)

5.7.1 The LRB is a reagent blank carried through the entire sample preparation process.

5.7.2 Employ a minimum of one laboratory reagent blank with each batch of 20 or fewer samples of the same matrix, to verify the absence of contamination. The LRB must be less than the reported detection limit for each analyte of interest.

5.8 Laboratory Fortified Blank (LFB)

5.8.1 A laboratory fortified blank (LFB) must be run with each sample batch. If the recovery falls outside the control limit of 80-120% or established control limits, whichever is more restrictive, the problem is to be identified and resolved before continuing.

5.8.2 The LFB is spiked prior to digestion using a source independent of both the standards and ICV and brought through the entire process.

5.9 Interference Check Solutions (ICS)

5.9.1 The ICS are analyzed in order to validate inter-element and background corrections applied to the samples.

5.9.2 The interference check solutions are prepared by combining known concentrations of interfering elements that will provide an adequate test of the correction factors, the “A fraction”.

5.9.3 Fortify the ICSAB solutions with the elements of interest in the 1 mg/L range, known as the “B fraction”.

5.9.4 In the absence of measurable analyte, over-correction could go undetected because a negative value could be reported as zero.

5.9.5 Analyze the ICSA and the ICSAB at the beginning and end of an analytical run or twice during every 8-hour work shift, whichever is more frequent. Recoveries of elements of interest should be within ±20% of the true values in the ICSAB and less than 2 times the reporting limit in the ICSA.

5.10 Sample Duplicate

5.10.1 Analyze one duplicate sample for every 20 samples. A duplicate sample is a sample brought through the entire sample preparation and analytical process. A control limit of ±20% for RPD shall be used for sample values greater than 10 times the instrument detection limit.

5.11 Laboratory Fortified Matrix / Duplicate (LFM/LFMD)

5.11.1 The LFM/LFMD pair must be run with each batch of 20 or fewer samples of the same matrix.

5.11.2 The LFM/LFMDs are prepared from fresh sample aliquots, spiked in the same manner as the LFB and carried through the entire preparation process.

5.11.3 The matrix spike and matrix spike duplicate spike recovery should be within ±25% of the true value, or documented control limits. Recovery calculations are not made if the spike concentration is less than 25% of the sample concentrations.

5.12 Inter-Element Corrections (IECs)

5.12.1 IECs are determined by analyzing a solution that contains an individual interfering element and is free of all other contaminates.

5.12.2 The positive or negative effects on the elements of interest are corrected by the following:

5.12.3 Correction value = true value of interfering element / concentration of the element of interest

5.12.4 IECs must only be evaluated and applied by analyst trained in there application.

5.12.5 IEC determination must be verified annually (at least) and updated, if necessary.

5.13 Linearity (L)

5.13.1 Dilute and reanalyze samples that are >90% of the established linear calibration limit or use an alternate, less sensitive line for which quality control data is established.

5.13.2 Linearity for all analytes must be updated quarterly.

5.14 Method Detection Limit (MDL)

5.14.1 MDLs must be maintained for each analyte of interest and updated once every year.

5.14.2 The determination of MDLs must be made in accordance with the following:

5.14.3 Fortify reagent water at a concentration of 2 to 3 times the estimated instrument detection limit.

5.14.4 Take seven replicate aliquots of the fortified reagent water and process through the entire analytical method.

5.14.5 Perform all calculations defined in the method and report the concentration values in the appropriate units.

5.14.6 Calculate the MDL as follows:

$$\mathbf{MDL = (t) \times (s)}$$

where: t = students' t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom [t = 3.143 for seven replicates].

S = standard deviation of the replicate analyses.

5.14.7 The final calculated MDL must be greater than 20% of the original analyte spike level.

5.15 Matrix Evaluation

5.15.1 It is recommended that whenever a new or unusual sample matrix is encountered, a series of tests be performed prior to reporting concentration data for analyte elements. These tests will ensure the analyst that neither positive nor negative interferences are operating on any of the analyte elements to distort the accuracy of the reported values. They are as follows:

5.15.2 Serial dilution

5.15.2.1 If the analyte concentration is sufficiently high (minimally, a factor of 10 above the instrumental detection limit after dilution), an analysis of a 1:5 dilution should agree within 10% of the original determination. If not, a chemical or physical interference effect should be suspected.

5.15.3 Post (digestion) Spike

5.15.3.1 An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 70% to 130% of the known value or the established control limits. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected. The use of a standard-addition analysis procedure may be used to compensate for this effect.

5.15.3.2 CAUTION: The standard-addition technique does not detect coincident spectral overlap. If suspected, use of computerized compensation (IECs), an alternate wavelength, or comparison with an alternate method is recommended.

5.16 Method of Standard Additions

5.16.1 The standard-addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume V_X, are taken. To the first (labeled A) is added a small volume V_S of a standard analyte solution of concentration C_S. To the second (labeled B) is added the same volume V_s of the solvent. The analytical signals of A and B are measured and corrected for non-analyte signals. The unknown sample concentration C_X is calculated

$$C_X = \frac{S_B * V_S * C_S}{}$$

$$(S_A - S_B) * V_X$$

where S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_S and C_S should be chosen so that S_A is roughly twice S_B on the average. It is best if V_S is made much less than V_X, and thus C_S is much greater than C_X, to avoid excess dilution of the sample matrix.

5.16.2 If a separation or concentration step is used, the additions are best made first and carried through the entire procedure. For the results of this technique to be valid, the following limitations must be taken into consideration:

a. The analytical curve must be linear, the correlation coefficient must be >0.995.

b. The chemical form of the analyte must respond the same way as the analyte in the sample.

c. The interference effect must be constant over the working range of concern.

d. The signal must be corrected for any additive interference.

5.16.3 The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated back to zero absorbance, the point of interception of the abscissa is the concentration of the unknown. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate.

VI. Calculations

6.1 Results are read in ug/L directly from the ICP. Take into account any dilutions preformed during the digestion process for total metals.

6.2 The recoveries of spikes and relative percent difference between duplicate determinations are to be calculated as follows:

$$\mathbf{RPD = \frac{|C_S - C_D|}{((C_S + C_D) / 2)}}$$

$$Rec = 100 * (C_M - C_S) / C_T$$

where RPD = relative percent difference, %
Rec = matrix spike recovery, %
C_S = unspiked sample concentration, mg/L
C_D = duplicate sample concentration, mg/mL
C_M = matrix spike concentration, mg/L
C_T = theoretical spike concentration, mg/L

Report recovery and RPD to the nearest 1 %.

VII. Reagents and Materials

7.1 Thermo Jarrell Ash 61E Simultaneous ICAP – capable of analysis and background correction for multi-element analysis

7.2 Thermo Jarrell Ash TRACE 61E Simultaneous ICAP - capable of trace analysis and background correction for multi-element analysis

7.3 Argon gas supply - high purity, liquid or high pressure cylinders

7.4 Concentrated hydrochloric acid - metals analysis grade

7.5 Hydrochloric acid, 1:1 dilution - add 500 mL concentrated hydrochloric acid to 400 mL reagent water and dilute to 1 liter

7.6 Concentrated nitric acid - Metals analysis grade

7.7 Nitric acid, 1:1dilution - add 500 mL concentrated nitric acid to 400 mL reagent water and dilute to 1 liter

7.8 Standard stock solutions - purchased from commercial suppliers

7.9 Second source solutions - purchased from commercial suppliers

7.10 Mixed calibration standard solutions

7.10.1 Prepare mixed calibration standard solutions by combining appropriate volumes of the stock solutions in volumetric.

7.10.2 Add the appropriate types and volumes of acids to match sample matrix. Care should be taken when preparing the mixed standards to ensure that the elements are compatible and stable together.

7.10.3 Transfer the mixed standard solutions to PFE fluorocarbon or previously unused polyethylene or polypropylene bottles for storage.

7.10.4 Fresh mixed standards should be prepared, as needed, with the realization that concentration can change on aging.

7.10.5 Calibration standards must be initially verified using a quality control sample and monitored for stability.

7.11 **Important:** If the addition of silver to the recommended acid combination results in an initial precipitation, add 15 mL of reagent water and warm the flask until the solution clears. Cool and dilute to 100 mL with reagent water. For this acid combination, the silver concentration should be limited to 2 mg/L. Silver under these conditions is stable in a tap-water matrix for 30 days. Higher concentrations of silver require additional hydrochloric acid.

7.12 If the sample analysis solution has a different acid concentration from that given, but does not introduce a physical interference or affect the analytical result, the same calibration standards may be used.

VIII. Safety

8.1 Every sample should be considered a hazardous when performing the analysis. Standard laboratory safety guidelines must be adhered to. Gloves, eye protection, and lab coats must be worn during sample retrieval, analysis and disposal.

IX. Pollution Prevention

9.1 Any and all remaining unused sample must be returned to the 4°C storage, sealed tightly in the original container. Benches and surrounding surfaces must be cleaned and wiped dry with paper toweling.

X. Waste management

10.1 Analyzed sample and used disposable equipment must be collected and disposed of in a manner consistent with the Premier Laboratory Chemical Hygiene Plan.

XI. Method Performance

11.1 Performance data is not currently available.

SOP Revision History

Date	Previous Revision No.	New Revision No.	Description of Changes	Effective Date	Initiated by
8/6/09	3.1	3.2	Added revision history table	8/6/09	LM
9/23/11	3.2	3.3	Changed format	9/23/11	LM

Mercury in Solids (Automated)
SW-846 7471A

Prepared by: Melisa Montgomery, Quality Assurance Officer — Approved by: Robert Stevenson, Laboratory Director

Reviewed and Implemented by: Ronald Warila, General Manager

Reference:

Test Methods for Evaluating Solid Waste, SW-846, Revision 1, September 1994, Method 7471A.

I. Applicability

1.1 Analyte: Mercury

1.2 Matrix: Soil, sludge, and waste extracts.

1.3 Regulation: RCRA

II. Important Notes

2.1 Method 7471A was developed to perform mercury in soil analysis via manual determination by cold vapor. Premier Laboratory employs the use of a Perkin Elmer FIMS 100 automated mercury analysis system. The FIMS 100 system was developed to replace manual determination of mercury by cold-vapor atomic absorption with an automated approach. Digestates are placed on the FIMS 100 autosampler where reagents are automatically added to the samples. The FIMS 100 software controls the addition of reagents, construction of the calibration curves, and the calculations for mercury determination in the samples.

2.2 During the digestion, potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/Kg of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from reagent water.

2.3 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/Kg had no effect on recovery of mercury from spiked samples.

2.4 Samples high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253 nm.

2.5 Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine hydrochloride reagent (25 mg/L).

2.6 Certain volatile organic materials that absorb at this wavelength may also cause interference. A preliminary run without reagents should determine if this type of interference is present.

2.7 The instrument method detection limit study must achieve a minimum detection of 0.005 mg/Kg.

2.8 The reporting limit for soils before dry weight adjustment is 0.02 mg/Kg.

2.9 The following cleaning sequence must be used for all glassware that will contact samples to be analyzed for metals:

a. Detergent wash
b. Tap water rinse
c. 1:1 nitric acid rinse
d. Reagent water rinse
e. 1:1 hydrochloric acid rinse
f. Reagent water rinse

III. Procedure

3.1 Transfer 1.0 g to 2.0g of sample to a 300 mL BOD bottle.

3.2 Add 10 mL of 1:1 Aqua Regia / Water, cover and transfer to a 95^{o}C water bath for 2 minutes.

3.3 Remove and allow to cool.

3.4 Add 50 mL of reagent water and 15 mL of $KMnO_4$.

3.4.1 Sewage samples and samples containing a high salt content may require additional portions of potassium permanganate solution.

3.4.2 If necessary, add 3.0 mL portions until the purple color persists for at least 15 minutes. Be sure to mix sample after each addition.

3.4.3 Track and record the additions to determine final volume.

3.5 Add 8 mL of potassium persulfate solution, return to the water bath for 30 minutes, covered.

3.6 Cool and add 6 mL of hydroxylamine hydrochloride solution and 50 mL of distilled water.

3.7 When the solution has been de-colorized, transfer to the autosampler.

3.8 All standards, quality control samples, and unknown samples are prepared at the same time and in the same manner.

3.9 Standard Concentrations

3.9.1 Intermediate Stock Solution:

3.9.1.1 Combine 0.1 mL of (1000 ppm) Hg stock, 5.0 mL H_2SO_4, 2.5 mL HNO_3, and 91.5 mL of reagent water in a 100 mL volumetric flask. The final concentration of the stock solution is 1000 ppb Hg.

3.9.2 Calibration standards:

3.9.2.1 All standards are prepared by transferring the appropriate aliquot of intermediate stock solution directly to digestion vessels. All standards are then carried through the entire digestion procedure.

3.9.3 The following is an example of dilutions made from a 1000ppm working solution into a 100 mL final volume.

Standards	Aliquot Added, mL	Final Concentration, ppb
STD 1 (blank)	0.0	0.0
STD 2	0.02	0.2
STD 3	0.1	1.0
STD 4	0.2	2.0
STD 5	0.5	5.0
STD 6	1.0	10.0

3.9.4 Proceed with calibration and analysis per FIMS 100 manual. The Zero Intercept Linear Calibration is represented by a straight line defined using the equation:

$$\mathbf{C = K_o(-K_1A)}$$

3.9.5 A calibration curve defined using this equation is forced to go through zero absorbance and zero concentration. A least squares technique is use to determine the K_1 coefficient when two or more standards are used for calibration. Ko is the re-slope coefficient, which is set to 1.0 during initial calibration.

3.9.6 Hg Analysis Sequence

Position	Sample Description
1 to 6	Calibration Standards 1 to 6
7	ICB
8	ICV
9	LRB (Laboratory Reagent Blank)
10	LFB (Laboratory Fortified Blank)
11	LLCS (Low Level Check Standard)
12	Sample 1
13	Sample 1 Duplicate
14 to 15	Sample 1 LFM/LFMD
16 to 20	5 Samples
21	CCB
22	Low CCV
23	Mid CCV
24 to33	10 Samples
34	CCV
35	CCB

3.9.6.1 Transfer the samples to the FIMS 100 autosampler and proceed with analysis per FIMS manual setup.

IV. Calculations

4.1 Direct reading in ug/L from the mercury autoanalyzer.

V. Quality Assurance

5.1 All quality control data should be maintained and available for easy reference or inspection.

5.2 Calibration Solutions

5.2.1 The calibration solutions are made using the same or similar acid matrix as the samples to be analyzed.

5.3 Initial Calibration Verification (ICV)

5.3.1 The ICV must be made from an outside second source different from that of the calibration standards' stock solutions.

5.3.2 The ICV is run immediately following the calibration and is used to verify calibration standards or stock solutions. The ICV recovery must be within ±10% of the true value for each analyte of interest.

5.4 Continuing Calibration Verification (CCV)

5.4.1 The CCV must be run periodically (every 10 samples) and at the end of each analytical sequence. The CCV is made from the same source as the calibration standards at mid-level concentration.

5.4.2 The recovery must be ±20% of the true value. The CCV may be run one additional time if the specified recoveries are not met, however if the second analysis fails, corrective action must be taken and any samples analyzed after the previous valid CCV must be re-analyzed.

5.5 Calibration Blank

5.5.1 The calibration blank contains the same acid matrix as the calibration standards and run with each ICV. The calibration blank is also used as the Continuing Calibration Blank (CCB) solution. See note 1. The criteria by which the blank results are to be evaluated is the following:

5.5.2 The results of the calibration blank are to agree within +/- the PQL. If not, repeat the analysis two more times and average the results. If the average is not within +/- the PQL, terminate the analysis, correct the problem; re-calibrate; and reanalyze the previous 10 samples.

5.6 Laboratory Reagent Blank (LRB)

5.6.1 The LRB is a reagent blank carried through the entire sample preparation process.

5.6.2 Employ a minimum of one laboratory reagent blank with each batch of 20 or fewer samples of the same matrix, to verify the absence of contamination. The LRB must be less than the reported detection limit.

5.7 Laboratory Fortified Blank (LFB)

5.7.1 A laboratory fortified blank (LFB) must be run with each sample batch. If the recovery falls outside the control limit of 80-120% or established control limits, the problem is to be identified and resolved before continuing.

5.8 Sample Duplicate

5.8.1 Analyze one duplicate sample for every 20 samples. A duplicate sample is a sample brought through the entire sample preparation and analytical process. A control limit of ±20% for RPD shall be used for sample values greater than 5 times the method detection limit. Samples less than 5 times the detection limit should be within +/- the method detection limit. Method 245.1 does not require the sample duplicate to be run, but it should remain an element of the normal QA protocol.

5.9 Laboratory Fortified Matrix / Duplicate (LFM/LFMD)

5.9.1 The LFM/LFMD pair must be run with each batch of 20 or fewer samples of the same matrix.

5.9.2 The LFM/LFMDs are prepared from fresh sample aliquots, spiked in the same manner as the LFB and carried through the entire preparation process.

5.9.3 The matrix spike and matrix spike duplicate spike recovery should be within ±25% of the true value, or documented control limits. Recovery calculations are not made if the spike concentration is less than 25% of the sample concentration.

5.10 Linear Dynamic Range (LDR)

5.10.1 Dilute and reanalyze samples that are >90% of the established linear calibration limit or use an alternate, less sensitive line for which quality control data is established.

5.10.2 Linear range should be determined at least every 6 months.

5.11 Method Detection Limit (MDL)

5.11.1 MDLs must be maintained for each analyte of interest and updated once every year.

5.11.2 The determination of MDLs must be made in accordance with the following:

5.11.3 Fortify reagent water at a concentration of 2 to 3 times the estimated instrument detection limit.

5.11.4 Take seven replicate aliquots of the fortified reagent water and process through the entire analytical method.

5.11.5 Perform all calculations defined in the method and report the concentration values in the appropriate units.

5.11.6 Calculate the MDL as follows:

$$\mathbf{MDL = (t) \times (s)}$$

where: t = students' t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom [t = 3.143 for seven replicates]

S = standard deviation of the replicate analyses

5.11.7 The final calculated MDL must be greater than 20% of the original analyte spike level.

5.12 Matrix Evaluation

5.12.1 It is recommended that whenever a new or unusual sample matrix is encountered, a series of tests be performed prior to reporting concentration data for analyte elements. These tests will ensure the analyst that neither positive nor negative interferences are operating on any of the analyte elements to distort the accuracy of the reported values. They are as follows:

5.12.2 Serial dilution

5.12.2.1 If the analyte concentration is sufficiently high (minimally, a factor of 10 above the instrumental detection limit after dilution), an analysis of a 1:4 dilution should agree within 10% of the original determination. If not, a chemical or physical interference effect should be suspected and a post spike must be run.

5.12.3 Post (digestion) Spike

5.12.3.1 An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 85% to 115% of the known value or the established control limits.

5.12.3.2 The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit.

5.12.3.3 If the spike is not recovered within the specified limits, a matrix effect should be suspected. The use of a standard-addition analysis procedure may be used to compensate for this effect.

VI. Reagents and Materials

6.1 Hg Analyzer – Perkin-Elmer FIMS 100 automated mercury system with associated software

6.2 Analysis vessels – 300 mL BOD bottles, 50 mL polypropylene centrifuge tubes

6.3 Hydrochloric acid – Concentrated, metals analysis grade

6.4 Sulfuric acid – Concentrated, metals analysis grade

6.5 Sulfuric acid solution, 0.5*N*

6.5.1 Dilute 14.0 mL of concentrated sulfuric acid to 1.0 liter

6.6 Nitric acid – Concentrated, metals analysis grade

6.7 Stannous chloride, (SnCl2)

6.7.1 Add 11 g stannous chloride to 1000 mL of 3% hydrochloric acid solution

6.8 Sodium chloride-hydroxylamine hydrochloride solution

6.8.1 Dissolve 12 g of sodium chloride (NaCl) and 24 g of hydroxylamine hydrochloride in reagent water and dilute to 100 mL

6.9 Potassium permanganate,($KMnO_4$), 5% w/v

6.9.1 Dissolve 50 g of potassium permanganate in 1000 mL of reagent water.

6.10 Potassium persulfate, ($K_2(SO_4)_2$), 5% w/v

6.10.1 Dissolve 50g of potassium persulfate in 1000 mL of reagent water.

6.11 Stock mercury standard, 1000 mg/L, purchased from a certified vendor

6.12 Mercury working standard

6.12.1 Make successive dilutions of the stock mercury standard to obtain a working standard.

6.12.2 The dilutions of the stock mercury standard must be prepared fresh daily.

6.12.3 Acidity of the working standard should be 2.5% nitric acid and 5% sulfuric acid.

6.12.4 This acid should be added to the volumetric flask before addition of the standard.

VII. Safety

7.1 Every sample should be considered a hazardous when performing the analysis. Standard laboratory safety guidelines must be adhered to. Gloves, eye protection, and lab coats must be worn during sample retrieval, analysis and disposal.

VIII. Pollution Prevention

8.1 Any and all remaining unused sample must be returned to the 4°C storage, sealed tightly in the original container. Benches and surrounding surfaces must be cleaned and wiped dry with paper toweling.

IX. Waste Management

9.1 Analyzed sample and used disposable equipment must be collected and disposed of in a manner consistent with the Premier Laboratory Chemical Hygiene Plan.

X. Method Performance

10.1 Performance data is not currently available.

SOP Revision History

Date	Previous Revision No.	New Revision No.	Description of Changes	Effective Date	Initiated by
12/11/09	2.2	2.3	Added revision history table	12/11/09	LM
4/29/11	2.3	2.4	Changed format	4/29/11	LM

Polychlorinated Biphenyls by Gas Chromatography
SW-846 8082

Prepared by: Melisa Montgomery, Quality Assurance Officer
Approved by: Robert Stevenson, Laboratory Director
Reviewed and Implemented by: Ronald Warila, General Manager

Reference

Test Methods for Evaluating Solid Waste, SW-846, Revision 0, December 1996, method 8082.

I. Applicability

1.1 Analyte: Polychlorinated biphenyls as Aroclors listed in Table 1.

1.2 Matrix: Extracts from solid waste matrices, soils, and aqueous samples

1.3 Regulation: RCRA

II. Important Notes

2.1 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of a solvent blank to check for cross contamination.

III. Summary

3.1 A measured volume or weight of sample (approximately 1 L for liquids, 2 g to 30 g for solids) is extracted using the appropriate matrix-specific sample extraction technique.

3.2 Liquid samples are extracted at neutral pH with methylene chloride using either Method 3510 (separatory funnel), Method 3520 (continuous liquid-liquid extractor), or other appropriate technique.

3.3 Solid samples are extracted with hexane, hexane-acetone (1:1) or methylene chloride-acetone (1:1) using Method 3545 (pressurized fluid extraction), Method 3550 (ultrasonic extraction), or other appropriate technique.

3.4 Extracts for PCB analysis may be subjected to a sulfuric acid/potassium permanganate clean up (Method 3665) designed specifically for these analytes. This cleanup technique will remove (destroy) many single component organochlorine or organophosphorus pesticides. Therefore, Method 8082 is not applicable to the analysis of those compounds. Instead, use Method 8081.

IV. Interferences

4.1 Refer to Methods 3500 (Sec. 3.0, in particular), 3600, and 8000, for a discussion of interferences

4.2 Sources of interference in this method can be grouped into three broad categories

a. Contaminated solvents, reagents, or sample processing hardware

b. Contaminated GC carrier gas, contaminated injection port, column surfaces, or detector surfaces

c. Compounds extracted from the sample matrix to which the detector will respond

4.3 Interferences by phthalate esters introduced during sample preparation can pose a major problem in PCB determinations.

a. These materials may be removed prior to analysis using Method 3665 (Sulfuric acid/permanganate cleanup).

b. Common flexible plastics contain varying amounts of phthalate esters, which are easily extracted or leached from such materials during laboratory operations.

c. Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled.

d. Interferences from phthalate esters can best be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

4.4 Glassware must be scrupulously cleaned.

4.4.1 Clean all glassware as soon as possible after use by rinsing with the last solvent used. This should be followed by detergent washing with hot water, and rinses with tap water and organic-free reagent water.

4.4.2 Drain the glassware and dry it in an oven at 130°C for several hours, or rinse with methanol and drain. Store dry glassware in a clean environment.

4.4.3 Do not dry glassware from samples containing high concentrations of PCBs with glassware that may be used for trace analysis due to the volatilization and spread of PCBs in the oven.

4.5 The presence of elemental sulfur will result in broad peaks that interfere with the detection of early eluting compounds. Sulfur contamination should be expected with sediment samples. Method 3660 is suggested for removal of sulfur.

V. Apparatus and Materials

5.1 Gas chromatograph/dual electron capture detectors/data system.

5.2 Chromatography columns: RTX-CLP Pesticides-1 (30m x 0.32 mm I.D. x 0.50 um)
RTX-CLP Pesticides-2 (30m x 0.32 mm I.D. x 0.25 um)

5.3 Syringes: 10µL, 25µL, 100µL, and 1000µL

VI. Reagents

6.1 Stock standard solutions: 1000mg/L. Purchased from commercial suppliers of certified standards.

6.1.1 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at -10 ^{0}C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.1.2 Stock standard solutions must be replaced after 1 year or sooner if comparison with quality control check samples indicates a problem.

6.2 Reagent grade or pesticide grade chemicals shall be used in all tests.

6.3 Calibration standards for Aroclors: A standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures. As a result, a multi-point initial calibration employing a mixture of Aroclors 1016 and 1260 at

five concentrations is used to demonstrate the linearity of the detector response without the necessity of performing initial calibrations for each of the seven Aroclors. In addition, the mixture is used to demonstrate that a sample does not contain peaks that represent any one of the Aroclors. This standard can also be used to determine the concentrations of either Aroclor 1016 or Aroclor 1260, should they be present in a sample.

6.3.1 A minimum of five calibration standards containing equal concentrations of both Aroclor 1016 and Aroclor 1260 by dilution of the stock standard with isooctane or hexane. Initial Calibration standards are prepared by diluting commercial standards to provide concentrations of 0.2, 1.0, 2.0, 5.0, and 10.0 mg/L.

6.3.2 All standards should be stored at -10 ^{0}C to -20 ^{0}C and should be freshly prepared once a year, or sooner if check standards indicate a problem. The daily calibration standard (2.0 mg/L) should be prepared weekly and stored at -10 ^{0}C to -20 ^{0}C.

6.4 Single standards of each of the other five Aroclors are required to aid the analyst in pattern recognition. Assuming that the Aroclor 1016/1260 standards described in the above section have been used to demonstrate the linearity of the detector, the single standards of the remaining five Aroclors are used to determine the calibration factor for each Aroclor. Prepare a standard for each of the other Aroclors at 2.0 mg/L, which corresponds typically to the mid-point of the linear range of the detector.

6.5 Surrogate Standards -The performance of the method is monitored using the surrogate compounds: Decachlorobiphenyl and Tetrachlo-m-xylene. Surrogate standards are added to all samples, method blanks, matrix spikes, and calibration standards. Control charts must be prepared and updated at least annually to define the surrogate acceptance ranges for all matrices.

VII. Procedure

7.1 Sample Preparation

7.1.1 Sample extraction

7.1.1.1 Refer to Chapter Two of SW-846 and Method 3500 for guidance in choosing the appropriate extraction procedure.

7.1.1.2 In general, water samples are extracted at a neutral pH with methylene chloride using a separatory funnel (Method 3510) or a continuous liquid-liquid extractor (Method 3520), or other appropriate technique.

7.1.1.3 Solid samples are extracted with hexane or hexane-acetone (1:1) using the pressurized fluid extraction (Method 3545), ultrasonic extraction (Method 3550), or other appropriate technique.

7.1.1.4 Extract cleanup- Refer to Methods 3660 (if a sulfur interference is observed in the preliminary scan), 3665, and 3620.

7.2 Suggested GC Operating Conditions

Column temperature program	180°C hold for 0 minutes Ramp to 240°C at 11°C/min, hold 0 min Ramp to 300°C at 20°C/min, hold 4.5 min
Detector temperature	300°C
Injector temperature	280°C
Injector	Grob-type, splitless
Sample volume	2.0 µL
Carrier gas	Argon/Methane P-5 mix
Column 1	RTX-CLP Pesticides-1 (30m x 0.32 mm I.D. x 0.50 um)
Column 2	RTX-CLP Pesticides-2 (30m x 0.32 mm I.D. x 0.25 um)

VIII. Initial Calibration

8.1 Because of the sensitivity of the electron capture detector, the injection port and column should always be cleaned prior to performing the initial calibration.

8.2 Calibration standards: Initial Calibration standards containing a mixture of Aroclor 1016 and 1260 are prepared by diluting commercial standards to provide concentrations of 0.2, 1.0, 2.0, 5.0, and 10.0 mg/L. A standard concentration of 0.1 mg/L may also be included if required to meet specific project detection limits. All standards are stored at -10 ^{0}C to -20 ^{0}C and should be freshly prepared once a year, or sooner if check standards indicate a problem.

8.3 A standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures. Thus, such a standard may be used to demonstrate the linearity of the detector and that a sample does not contain peaks that represent any one of the Aroclors. This standard can also be used to determine the concentrations of either Aroclor 1016 or Aroclor 1260, should they be present in a sample.

Therefore, an initial five-point calibration is performed using the mixture of Aroclors 1016 and 1260.

8.4 Standards of the other five Aroclors are necessary for pattern recognition. These standards are also used to determine a single-point calibration factor for each Aroclor, assuming that the Aroclor 1016/1260 mixture has been used to describe the detector response. The standards for these five Aroclors should be analyzed before the analysis of any samples, and may be analyzed before or after the analysis of the five 1016/1260 standards.

8.5 Unless otherwise necessary for a specific project, the analysis of the multi-component analytes employs a single point calibration. A single calibration standard near the mid point of the expected calibration range of each multi-component analyte is included with the initial calibration of the single component analytes for pattern recognition, so that the analyst is familiar with the patterns and retention times on each column.

8.6 A minimum of 3 peaks must be chosen for each Aroclor. The peaks must be characteristic of the Aroclor in question. Choose peaks in the Aroclor standards that are at least 25% of the height of the largest Aroclor peak. For each Aroclor, the set of 3 peaks must include at least one peak that is unique to that Aroclor. Use 6 peaks (3 for 1016, 3 for 1260) for the Aroclor 1016/1260 mixture, none of which should be found in both of these Aroclors.

8.7 A 2 µL injection volume of each calibration standard is recommended. Other injection volumes may be employed, provided that the analyst can demonstrate adequate sensitivity for the compounds of interest.

8.8 External standard calibration: Calculate the calibration factor for each characteristic Aroclor peak at each concentration, the mean calibration factor, and the relative standard deviation (RSD) of the calibration factors, using the formula below.

8.9 Calculate the calibration factor for each characteristic peak at each concentration:

$$\mathbf{CF = \frac{\text{Peak Area of the Compound in the Standard}}{\text{Mass of the Compound Injected (in nanograms)}}}$$

8.10 Calculate the mean calibration factor for each peak.

8.11 Calculate the relative standard deviation (RSD) of the calibration factors for each peak.

8.12 Five sets of calibration factors will be generated for the Aroclor 1016/1260 mixture, each set consisting of the calibration factors for each of the 6 peaks chosen for this mixture. The single standard for each of the other Aroclors will generate at least three calibration factors, one for each selected peak.

8.13 If the RSD for each peak of the 1016/1260 mixture is <20%, then the response of the instrument is considered linear and the mean calibration factor can be used to quantitate sample results. If the RSD is greater than 20%, then linearity through the origin cannot be assumed. The analyst must use a calibration curve or a non-linear calibration model (e.g. a polynomial equation) for quantitation.

8.14 The mean calibration factor is acceptable for quantitating sample results when meeting the quality objectives for specific projects if the following is met.

8.15 The mean of the RSDs for the 3 peaks of each compound are less than 20% while allowing 1 peak to exceed individually the 20% RSD criteria.

8.16 This criteria must be acceptable and directed by the data end user prior to its application.

IX. Continuing Calibration and Sample Analysis:

9.1 The same GC operating conditions used for the initial calibration must be employed for sample analyses.

9.2 Verify calibration each 12-hour shift by injecting a calibration verification standard of Aroclor 1016/1260 prior to conducting any sample analyses. A calibration standard must also be injected at intervals of not less than once every twenty samples (after every 10 samples is *recommended* to minimize the number of samples requiring re-injection when QC limits are exceeded) and at the end of the analysis sequence.

9.3 The calibration factor for each analyte must not exceed a $\pm$ 15 percent difference from the mean calibration factor calculated for the initial calibration.

$$\% \text{ Difference} = \frac{CF - CF_{AVG}}{CF_{AVG}} \times 100$$

9.4 If the calibration does not meet the ±15% limit, check the instrument operating conditions, and if necessary, restore them to the original settings, and inject another aliquot of the calibration verification standard. If the response for the analyte is still not within ±15%, then a new initial calibration must be prepared.

9.5 Inject a 2-µL aliquot of the concentrated sample extract. Record the volume injected to the nearest 0.05 µL and the resulting peak size in area units.

X. Quantitation

10.1 Calculate the sample concentration using the mean CF from the initial calibration.

10.1.1 For aqueous samples:

$$\textbf{Concentration (}\mu\textbf{g/L)} = \frac{(A_X)(V_T)(D)}{(CF_{AVG})(V_i)(V_S)}$$

where: A_X = Area of the peak for the analyte in the sample

V_T = Total volume of the concentrated extract (µL)

D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution was made, D is equal to 1. The dilution factor is always dimensionless.

CF = Mean calibration factor from the initial calibration (area/ng)

V_i = Volume of the extract injected (µL). The injection volume for samples and calibration standards must be the same

V_S = Volume of the aqueous sample extracted in mL. If units of liters are used for this term, multiply the results by 1000

10.1.1.1 Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to µg/L.

10.1.2 For non-aqueous samples:

$$\textbf{Concentration (}\mu\textbf{g/kg)} = \frac{(A_X)(V_T)(D)}{(CF_{AVG})(V_i)(W_S)(TS)}$$

where: A_X, V_T, D, CF, and V_i are the same as for aqueous samples,

W_S = Weight of sample extracted (g)

TS = % solids in sample (to report on a dry weight basis)

10.2 If the responses exceed the calibration range of the system, dilute the extract and reanalyze. Peak height measurements are recommended over peak area integration when overlapping peaks cause errors in area integration.

10.3 When simultaneous analyses are performed from a single injection, it is not practical to designate one column as the analytical (primary) column and the other as the confirmation column. Since the calibration standards are analyzed on both columns, the results for both

columns must meet the calibration acceptance criteria. If the retention times of the peaks on both columns fall within the retention time windows on the respective columns, then the target analyte identification has been confirmed.

10.4 Each sample analysis must be bracketed with an acceptable initial calibration, calibration verification standard(s) (each 12-hour analytical shift), or calibration standards interspersed within the samples.

10.5 The results from these bracketing standards must meet the $\pm$ 15% continuing calibration verification criteria. When a calibration verification standard fails to meet the QC criteria, all samples that were injected after the last standard that last met the QC criteria must be evaluated to prevent mis-quantitations and possible false negative results, and re-injection of the sample extracts may be required. More frequent analyses of standards will minimize the number of sample extracts that would have to be reinjected if the QC limits are violated for the standard analysis.

10.6 Sample injections may continue for as long as the calibration verification standards and standards interspersed with the samples meet instrument QC requirements. It is *recommended* that standards be analyzed after every 10 samples *(required* after every 20 samples and at the end of a set) to minimize the number of samples that must be re-injected when the standards fail the QC limits. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded.

10.7 The quantitation of PCB residues as Aroclors is accomplished by comparison of the sample chromatogram to that of the most similar Aroclor standard. A choice must be made as to which Aroclor is most similar to that of the residue and whether that standard is truly representative of the PCBs in the sample.

10.8 Use the individual Aroclor standards (not the 1016/1260 mixtures) to determine the pattern of peaks on Aroclors 1221,1232,1242,1248, and 1254. The patterns for Aroclors 1016 and 1260 will be evident in the mixed calibration standards.

10.9 Once the Aroclor pattern has been identified, compare the responses of 3 to 5 major peaks in the single-point calibration standard for that Aroclor with the peaks observed in the sample extract. The amount of Aroclor is calculated using the individual calibration factor for each of the 3 to 5 characteristic peaks chosen and the calibration model (linear or non-linear) established from the multi-point calibration of the 1016/1260 mixture. A concentration is determined using each of the characteristic peaks and then those 3 to 5 concentrations are averaged to determine the concentration of that Aroclor.

XI. Quality Control

11.1 Refer the Premier Laboratory Quality Manual for specific quality control procedures to demonstrate the ability to generate data of acceptable accuracy and precision for the method. This includes but is not limited to the following:

11.1.1 A method detection limit (MDL) study must be completed before samples can be analyzed. The MDL study must be repeated whenever a significant change in the procedure is made.

11.1.2 A initial demonstration of capability (IDC) must be completed by each qualified analyst before samples can be analyzed. The IDC must be repeated whenever a significant change in the procedure is made.

11.2 Method Blank Analysis

a) A method blank must be prepared and analyzed at a frequency of 1 per batch of samples extracted per matrix, not to exceed 20 samples.

b) The concentration of each target compound found in the blank must be less than the required quantitation limit for the project.

c) A solvent blank should be analyzed whenever a new lot of solvent is introduced to check for potential contamination.

11.3 Matrix Spike/ Laboratory Control Spike Analysis

11.3.1 A matrix spike and matrix spike duplicate pair must be extracted and analyzed at a frequency of 1 per 20 samples extracted per matrix.

11.3.2 One MS/MSD pair must be extracted and analyzed at least every 30 days for each matrix.

11.3.3 The laboratory must generate MS/MSD recovery data control charts at least annually for each matrix.

11.3.4 The control charts will be used to define acceptable recovery ranges of spike compounds.

11.3.5 If one or more compounds are outside of the control limits then an LCS (QC check standard) must be analyzed to check for matrix interference.

11.3.6 The LCS must meet the laboratory generated acceptance criteria for those compound that failed acceptance criteria in the MS/MSD.

11.3.7 Matrix spike recovery calculation:

$$\% \text{ Recovery} = \frac{\text{spiked sample result - sample result}}{\text{spike added}} \times 100$$

11.3.8 LCS recovery calculation:

$$\% \text{ Recovery} = \frac{\text{LCS sample result}}{\text{spike added}} \times 100$$

11.4 Surrogate Recoveries

11.4.1 Surrogate spike recovery limits must be generated and updated at least annually for each matrix through the use of control charts.

11.4.2 If the recovery of one or more compounds is outside of the control limits, the sample must be re-analyzed. If after re-analysis the recovery is still not within the limits the sample must be re-extracted and re-analyzed. If the re-extracted sample surrogates do not meet the criteria, then the matrix interference problem must be noted in the project case narrative or non-conformance summary.

Table 1

CAS No.	Compound	Soil Estimated Quantitation Limits, ug/kg	Aqueous Estimated Quantitation Limits, ug/L
12674-11-2	Aroclor 1016	13.3	0.4
11104-28-2	Aroclor 1221	13.3	0.4
11141-16-5	Aroclor 1232	13.3	0.4
53469-21-9	Aroclor 1242	13.3	0.4
12672-29-6	Aroclor 1248	13.3	0.4
11097-69-1	Aroclor 1254	13.3	0.4
11096-82-5	Aroclor 1260	13.3	0.4

SOP Revision History

Date	Previous Revision No.	New Revision No.	Description of Changes	Effective Date	Initiated by
8/6/09	1.1	1.2	Added revision history table	8/6/09	LM
9/23/11	1.2	1.3	Changed format	9/23/11	LM

Extractable Petroleum Hydrocarbons by GC/FID
MADEP EPH

Prepared by: Melisa Montgomery, Quality Assurance Officer
Approved by: Robert Stevenson, Laboratory Director
Reviewed and Implemented by: Ronald Warila, General Manager

References:

Method for the Determination of Extractable Petroleum Hydrocarbons (EPH), Mass. Dept. of Environmental Protection, May 2004, Revision 1.1

Recommended Reasonable Confidence Protocols, Quality Assurance and Quality Control Requirements Extractable Petroleum Hydrocarbons by the Massachusetts DEP EPH Method, State of Connecticut Dept. of Environmental Protection, Version 2.0, May 2009

I. Scope and Application

1.1 This method is designed to measure the collective concentrations of extractable aliphatic and aromatic petroleum hydrocarbons in water and soil/sediment matrices. Extractable aliphatic hydrocarbons are collectively quantitated within two ranges: C_9 through C_{18} and C_{19} through C_{36}. Extractable aromatic hydrocarbons are collectively quantitated within the C_{11} through C_{22} range. These aliphatic and aromatic hydrocarbon ranges correspond to a boiling point range between approximately 150 °C and 265 °C.

1.2 This method is based on a solvent extraction, silica gel solid-phase extraction (SPE)/fractionation process, and gas chromatography (GC) analysis using a flame ionization detector (FID). This procedure should be used by, or under the supervision of, analysts experienced in extractable organics analysis. Analysts should be skilled in the interpretation of gas chromatograms and their use as a quantitative tool.

1.3 This method is designed to complement and support the toxicological approach developed by the Massachusetts Department of Environmental Protection to evaluate human health hazards that may result from exposure to petroleum hydrocarbons (MADEP, 1994 and MADEP, 2003). It is intended to produce data in a format suitable for evaluation by that approach and that may be compared to reporting and cleanup standards promulgated in the Massachusetts Contingency Plan (310 CMR 40.0000).

1.4 This method is also able to measure the individual concentrations of target polynuclear aromatic hydrocarbons (PAH) analytes, including diesel PAH analytes, in water and soil/sediment matrices. The use of this method to quantify these analytes is optional, and the reporting limits for some of these PAH compounds in water are greater than the notification

and/or cleanup standards specified in the Massachusetts Contingency Plan for sites located in groundwater resource area categorized as RCGW-1 in 310 CMR 40.0362(1)(a). In cases where it is necessary to demonstrate compliance with these standards, the use of a gas chromatography/mass spectrometry (GC/MS) method in the selective ion monitoring (SIM) mode and/or high performance liquid chromatography (HPLC) methodology may be necessary.

1.5 The fractionation step described in this method can be eliminated to allow for a determination of a total petroleum hydrocarbon (TPH), and/or to obtain qualitative "fingerprinting" information. While TPH provides little information on the chemical constituents, toxicity, or environmental fate of petroleum mixtures, it may be a cost-effective screening tool in cases where relatively low concentrations of contamination are suspected.

1.6 Petroleum products suitable for evaluation by this method include kerosene, fuel oil #2, fuel oil #4, fuel oil #6, diesel fuel, jet fuel, and certain lubricating oils. This method, in and of itself, is not suitable for the evaluation of gasoline, mineral spirits, petroleum naphthas, or other petroleum products which contain a significant percentage of hydrocarbons lighter than C_9. This method, in and of itself, is also not suitable for the evaluation of petroleum products which contain a significant percentage of hydrocarbons heavier than C_{36}.

1.7 The reporting limit (RL) of this method for each of the collective aliphatic and aromatic fractional ranges is approximately 20 mg/kg in soil/sediment, and approximately 100 µg/L in water. The RL of this method for TPH is approximately 10 mg/kg in soil and approximately 100 µg/L in water. The RL of this method for the target PAH analytes is compound-specific, and ranges from approximately 0.2 to 1.0 mg/kg in soil/sediment, and 2 to 5 µg/L in water.

1.8 This method includes a data adjustment step to subtract the concentration of target PAH analytes from the concentration of C_{11} through C_{22} aromatic hydrocarbons.

1.9 Data reports produced using this method must contain all of the required EPH/TPH data information provided in Appendix 3 of the method. The format of these data reports is left to the discretion of individual laboratories.

1.10 Like all GC procedures, this method is subject to a "false positive" bias in the reporting of target PAH analytes, in that non-targeted hydrocarbon compounds eluting or co-eluting within a specified retention time window may be falsely identified and/or quantified as a target or diesel PAH analyte. In addition, this method is subject to a "false negative" bias in the reporting of target PAH analytes, in that the ability to identify target PAH analytes at low concentrations may be inhibited if a large unresolved complex mixture is present. While cleanup procedures specified in this method to segregate aliphatic and aromatic fractions will serve to mitigate these concerns, confirmatory analysis by dissimilar columns, GC/MS analysis, or other suitable technique is recommended in cases where a target PAH analyte reported by this method approaches or exceeds an applicable reporting or cleanup standard, and/or where co elution of a non-targeted hydrocarbon compound is suspected.

1.11 This method is one way to quantify collective concentrations of extractable aliphatic and aromatic petroleum hydrocarbons within specified carbon-number-ranges. It has been designed in a manner that attempts to strike a reasonable balance between analytical method performance and utility. In this manner, assumptions and biases have been incorporated into the method to help ensure protective, though not overly conservative data.

1.12 As an example, the Department recognizes that branched alkanes have lower boiling points than their n-alkane counterpart does, while many of the cycloalkane constituents of diesel range volatile organics have higher boiling points than their n-alkane counterpart. As a consequence:

a. Depending upon the specific chromatographic column used, most branched C_9 alkanes are expected to elute before n-nonane, the beginning marker compound for the C_9 through C_{18} aliphatic hydrocarbon range, and will not be counted in this range;

b. Depending upon the specific chromatographic column used, most branched C_{19} alkanes are expected to elute before n-nonadecane, the beginning marker compound for the C_{19} through C_{36} aliphatic hydrocarbon range, and will be conservatively counted in the more toxic C_9 through C_{18} aliphatic hydrocarbon range; and

c. Depending upon the specific chromatographic column used, most cycloalkanes within the C_9 through C_{18} and C_{19} through C_{36} aliphatic hydrocarbon ranges will be counted within their proper range.

1.13 Based on the nature of petroleum releases encountered in the environment, the collective concentrations of the extractable aliphatic ranges as measured by the EPH Method are considered to be suitable for the evaluation of the risks posed by these releases, consistent with the toxicological approach developed by the Department to evaluate human health hazards that may result from exposure to petroleum hydrocarbons (MADEP, 1994 and MADEP, 2003).

1.14 This method is used in conjunction with the current version of WSC-CAM-IV B, "Quality Assurance and Quality Control Requirements for the Method for the Determination of Extractable Petroleum Hydrocarbons (EPH)".

II. Summary of Method

2.1 A sample submitted for EPH analysis is extracted with methylene chloride, dried over sodium sulfate, solvent exchanged into hexane, and concentrated in a Turbovap apparatus. Sample cleanup and separation into aliphatic and aromatic fractions is accomplished using commercially available silica gel cartridges or prepared silica gel columns. The two individual fraction extracts produced are re-concentrated to a final volume of 1 mL (i.e., an aliphatic extract and an aromatic extract). The concentrated extracts are then separately analyzed by a capillary column gas chromatograph equipped with a flame ionization detector. The resultant chromatogram of aliphatic compounds is collectively integrated within the C_9 through C_{18} and C_{19} through C_{36} ranges. The resultant chromatogram of aromatic compounds is collectively integrated within the C_{11} through C_{22} range, and is (optionally) used to identify and quantitate individual concentrations of target PAH analytes.

2.2 Average calibration factors or response factors determined using an aliphatic hydrocarbon standard mixture are used to calculate the collective concentrations of C_9 through C_{18} and C_{19} through C_{36} aliphatic hydrocarbons. An average calibration factor or response factor determined using a PAH standard mixture is used to calculate a collective C_{11} through C_{22} aromatic hydrocarbon concentration. Calibration factors or response factors determined for individual components of the PAH standard mixture are also used to calculate individual concentrations of Target PAH Analytes.

2.3 This method is suitable for the analysis of waters, soils, sediments, wastes, sludges, and non-aqueous phase liquids (NAPL). However, it should be noted that the method was validated only for soil and water matrices.

2.4 This method is based on (1) USEPA Methods 8000B, 8100, 3510C, 3520C, 3540C, 3541, 3545A, 3546, 3580 A and 3630C, SW-846, "Test Methods for Evaluating Solid Waste"; (2) Draft "Method for Determination of Diesel Range Organics", EPA UST Workgroup, November, 1990; and (3) "Method for Determining Diesel Range Organics", Wisconsin Department of Natural Resources, PUBL-SW-141, 1992

III. Definitions

3.1 Aliphatic Hydrocarbon Standard is defined as a 14 component mixture of the normal alkanes listed in Table 1. The compounds comprising the aliphatic hydrocarbon standard are used to (a) define and establish windows for the two aliphatic hydrocarbons ranges, and (b) determine average calibration or response factors that can in turn be used to calculate the collective concentration of aliphatic hydrocarbons in environmental samples within those hydrocarbon ranges.

3.2 Analytical Batch is defined as a group of field samples with similar matrices which are processed as a unit. For quality control purposes, if the number of samples in such a group is greater than 20, then each group of 20 samples or less is defined as a separate analytical batch.

3.3 Aromatic Hydrocarbon Standard is defined as a 17 component mixture of the polynuclear aromatic hydrocarbons (PAHs) listed in Table 2 of the method. The compounds comprising the Aromatic Hydrocarbon Standard are used to: (a) define the individual retention times and calibration or response factors for each of the PAH analytes listed in Table 2, (b) define and establish the window for the C_{11} through C_{22} aromatic hydrocarbon range, and (c) determine an average calibration or response factor that can in turn be used to calculate the collective concentration of aromatic hydrocarbons in environmental samples within the C_{11} through C_{22} hydrocarbon range.

3.4 C_9 through C_{18} Aliphatic Hydrocarbons are defined as all aliphatic hydrocarbon compounds which contain between nine and 18 carbon atoms and are associated with the release of a petroleum product to the environment. In the EPH method, C_9 through C_{18} aliphatic hydrocarbons are defined and quantitated as compounds that elute from n-nonane (C_9) to just before n-nonadecane (C_{19}).

3.5 C_{19} through C_{36} Aliphatic Hydrocarbons are defined as all aliphatic hydrocarbon compounds which contain between 19 and 36 carbon atoms and are associated with the release of a petroleum product to the environment. In the EPH method, C_{19} through C_{36} aliphatic hydrocarbons are defined and quantitated as compounds that elute from n-nonadecane (C_{19}) to just after hexatriacontane (C_{36}).

3.6 C_{11} through C_{22} Aromatic Hydrocarbons are defined as all aromatic hydrocarbon compounds which contain between 11 and 22 carbon atoms and are associated with the release of a petroleum product to the environment. In the EPH method, C_{11} through C_{22} aromatic

hydrocarbons are defined and quantitated as compounds that elute from naphthalene to just after benzo(g,h,i)perylene, excluding target PAH analytes.

3.7 Calibration Standards are defined as a series of standard solutions prepared from dilutions of a stock standard solution, containing known concentrations of each analyte and surrogate compound of interest.

3.8 Continuing Calibration Standard is defined as a calibration standard used to periodically check the calibration state of an instrument. The continuing calibration standard is prepared from the same stock standard solution as calibration standards, and is generally one of the mid-level range calibration standard dilutions.

3.9 Diesel PAH Analytes are defined as naphthalene, 2-methylnaphthalene, phenanthrene, and acenaphthene, and are a subset of target PAH analytes. For most sites known to be contaminated by a release of diesel and/or #2 fuel oil only, diesel PAH analytes will be the only target PAH analytes of interest.

3.10 Extractable Petroleum Hydrocarbons (EPH) are defined as collective fractions of hydrocarbon compounds eluting from n-nonane to n-hexatriacontane, excluding target PAH analytes. EPH is comprised of C_9 through C_{18} aliphatic hydrocarbons, C_{19} through C_{36} aliphatic hydrocarbons, and C_{11} through C_{22} aromatic hydrocarbons.

3.11 Field Duplicates are defined as two separate samples collected at the same time and place under identical circumstances and managed the same throughout field and laboratory procedures. Analyses of field duplicates give a measure of the precision associated with sample collection, preservation and storage, as well as laboratory procedures.

3.12 Fractionation Surrogate Standards are compounds that are added to sample extracts immediately prior to fractionation at known concentrations to evaluate fractionation efficiency.

3.13 Initial Calibration Verification (ICV) Standard is defined as a mid-range standard prepared from a separate source than used for the initial and continuing calibration standards. The analysis of an ICV must be performed when a separate source standard is not used for the preparation of the laboratory control sample and matrix spike sample.

3.14 Laboratory Control Sample (LCS) is defined as a reagent water blank (when associated with aqueous samples) or clean sand blank (when associated with soil/sediment samples) fortified with a matrix spiking solution. The LCS is prepared and analyzed in the same manner as the samples and its purpose is to determine the bias of the analytical method.

3.15 Laboratory Control Sample Duplicate (LCSD) is defined as a reagent water blank (when associated with aqueous samples) or clean sand blank (when associated with soil/sediment samples) fortified with a matrix spiking solution separately prepared, processed and analyzed in the same manner as the LCS. The analysis of LCSD gives a measure of the precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

3.16 Laboratory Method Blank is defined as an aliquot of reagent water (when associated with aqueous samples) or clean sand (when associated with soil/sediment samples) spiked with a surrogate standard. The laboratory method blank is prepared and analyzed in the same

manner as a sample, exposed to all glassware, solvents, reagents, and equipment. A laboratory method blank is prepared and analyzed with every batch of samples, to determine if method analytes or other interferences are present in the laboratory environment, reagents, or equipment.

3.17 Matrix Duplicates are defined as split samples prepared and analyzed separately with identical procedures. For soil/sediment samples, matrix duplicate samples are taken from the same sampling container. For aqueous samples, a separate container is used for the matrix duplicate sample. The analysis of matrix duplicates gives a measure of the precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

3.18 Matrix Spike (MS) Sample is defined as an environmental sample which has been spiked with a matrix spiking solution containing known concentrations of method analytes. The purpose of the MS sample is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined through the separate analyses of an unspiked sample aliquot. The measured values in the MS sample must be corrected for background concentrations when calculating recoveries of spiked analytes.

3.19 Matrix Spiking Solution is defined as a solution prepared from a separate source than used for the calibration standards, containing known concentrations of method analytes.

3.20 System Solvent Blank is defined as an aliquot of a method solvent (e.g., hexane or methylene chloride, pesticide-grade or better) that is directly injected into the GC system. The system solvent blank provides one way of determining the level of noise and baseline rise attributable solely to the analytical system, in the absence of any other analytes or non-analytical related contaminants.

3.21 Surrogate Standards are compounds spiked into all samples, blanks, LCSs, and matrix spikes to monitor the efficacy of sample extraction, chromatographic, and calibration systems.

3.22 Target PAH Analytes are defined as the 17 polynuclear aromatic hydrocarbon (PAH) compounds listed in Table 2 of the method.

3.23 Total Petroleum Hydrocarbons (TPH) are defined as the collective concentration of all hydrocarbon compounds eluting from n-nonane to n-hexatriacontane, excluding target PAH analytes. TPH is equivalent to the summation of C_9 through C_{18} aliphatic hydrocarbons, C_{19} through C_{36} aliphatic hydrocarbons, and C_{11} through C_{22} aromatic hydrocarbons.

3.24 Unadjusted C_{11} through C_{22} Aromatic Hydrocarbons are defined as all aromatic hydrocarbon compounds eluting from naphthalene through benzo(g,h,i)perylene.

3.25 Unadjusted TPH is defined as the collective concentration of all hydrocarbon compounds eluting from n-nonane to n-hexatriacontane, including the target PAH analytes.

3.26 All other terms are as defined in the most current version of SW-846, "Test Method for Evaluating Solid Waste", USEPA.

IV. Interferences

4.1 Method interferences are reduced by washing all glassware with hot soapy water and then rinsing with warm tap water, acetone, and methylene chloride.

4.2 High purity reagents must be used to minimize interference problems.

4.3 Cross-contamination can occur whenever a low-concentration sample is analyzed immediately after a high-concentration sample. To reduce carryover, the sample syringe must be rinsed between samples with solvent. Whenever an unusually concentrated sample is encountered, it must be followed by the analysis of a system solvent blank to check for cross-contamination. However, due to the potential for samples to be analyzed using an auto sampler, the ability to perform this blank analysis may not always be possible. If the sample analyzed immediately after the unusually concentrated sample is free from contamination, then the assumption can be made that carryover or cross-contamination is not an issue. However, if this sample did detect analytes which were present in the unusually concentrated sample, reanalysis is required for all samples analyzed after this highly concentrated sample which detected similar analytes.

4.4 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interference will vary considerably from one source to another depending upon the nature and complexity of the site being sampled. A silica gel SPE cleanup procedure is used to overcome many of these interferences, but some samples may require additional and more rigorous cleanup procedures which are beyond the scope of this method.

4.5 Other organic contaminants commingled with petroleum product releases, including chlorinated hydrocarbons, phenols, and phthalate esters, will be quantitated as total and extractable petroleum hydrocarbons. If necessary and/or desirable, additional sample cleanup and/or analytical procedures may be employed to minimize or document the presence of such compounds.

4.6 The leaching of plasticizers and other compounds have been observed from commercially available silica gel cartridges used to fractionate EPH sample extracts. Concerns of this nature must be continuously monitored and documented by analysis of laboratory method blanks. Section 9.4 provides a procedure to eliminate or minimize this contamination.

4.7 Because of their weakly polar nature, naphthalene and substituted naphthalenes readily mobilize into the aliphatic extract if excessive amounts of hexane are used to elute the silica gel cartridge/column. Because these compounds constitute a significant percentage of the water-soluble fraction of fuel oils, this occurrence is especially problematic in the analysis of water samples. For this reason, the method requires the evaluation of the aliphatic fraction for the presence of naphthalene and 2-methylnaphthalene in the LCS/LCSD pair on a batch basis. The fractionation surrogate, 2-bromonaphthalene, is used to monitor sample-specific fractionation efficiency.

V. Health and Safety Issues

5.1 The toxicity and carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current

file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDS) should also be made available to all personnel involved in the chemical analysis.

VI. Apparatus and Materials

6.1 The following is a partial list of glassware used for this method:

a. 1-L amber glass bottles
b. 4 oz. (120 mL) amber wide-mouth glass jars
c. 2-mL glass auto sampler vials with Teflon-lined rubber crimp caps
d. 10-mL vials with Teflon-lined caps
e. Glass funnels
f. 2-L Separatory funnels with Teflon stopcock (aqueous liquid-liquid extraction only)
g. 250-mL Erlenmeyer flasks
h. 25-mL graduated cylinder
i. 1-Liter graduated cylinder
j. 100-mL beakers
k. Class "A" volumetric flasks: 10, 25, 50 and 100-mL
l. Class "A" volumetric pipets: 1, 5 or 10-mL

6.2 An analytical balance capable of accurately weighing 0.0001 g must be used for weighing standards. A top-loading balance capable of weighing to the nearest 0.1 g must be used for weighing soil/sediment samples.

6.3 An air or nitrogen blow down apparatus, or equivalent sample concentration apparatus, is required to concentrate extracts.

6.4 Gas Chromatographic System: An analytical system incorporating a temperature-programmable oven with the ability to accommodate a capillary column. The following components are also required:

a. Detector: A Flame Ionization Detector (FID) is required.
b. Column: The analytical column must adequately resolve the n-C_9 to n-C_{36} aliphatic hydrocarbon standard compounds and the target PAH analytes listed in Tables 1 and 2, respectively. The recommended analytical column is an RXi-5MS capillary column (30-m x 0.32-mm i.d., 0.25-µm film thickness [Restek Corp. part number 13624 or equivalent]).
c. Data Station: The data station must be capable of storing and reintegrating chromatographic data and must be capable of determining peak areas using a forced baseline projection.
d. Auto sampler: An auto sampler capable of making 1 to 4 µL injections is recommended.

6.5 Disposable pipets: Pasteur

6.6 Micro syringes: 10-µL, 100-µL, 250-µL, 500-µL, 1000-µL

6.7 Drying oven

6.8 Desiccators

VII. Reagents and Standards

7.1 Reagents

a. Reagent Water: organic free water (ASTM Type I reagent grade water).

b. Solvents: hexane, methylene chloride, and acetone; pesticide-grade or better. Store away from other solvents.

c. Sodium sulfate: (ACS) granular, anhydrous. Purify by heating at 400°C for 4 hours in a shallow tray.

d. Ottawa and/or masonry sand: free of extractable petroleum hydrocarbons.

e. Diatomaceous earth: free of extractable petroleum hydrocarbons.

f. Silica Gel (5 - 10 grams), either prepared and packed by the laboratory, or purchased in 5 g/20-mL cartridges from a commercial vendor (Restek Corp. part no. 26065). Silica gel prepared and packed by the laboratory should be activated at 130 ºC for at least 16 hours, and heated to 150-160 ºC for several hours before use.

Important: Leaching of plasticizers and other compounds have been observed from commercially prepared silica gel cartridges, and must be monitored and documented by analyses of Laboratory Method Blanks.

Silica gel is hygroscopic. Unused cartridges readily absorb moisture from ambient air if not properly sealed. To preclude moisture adsorption, which adversely effects cartridge performance, unused cartridges must be stored in a properly maintained desiccator prior to use.

7.2 Stock Standard Solutions: Prepare stock standard solutions at approximately 100 mg/L, or purchase as certified solutions. See Table 2 for details on standard solutions.

7.2.1 Aromatic Hydrocarbon Standard: The aromatic hydrocarbon standard consists of the 17 PAH compounds listed in Table 2, a surrogate compound (i.e., ortho-terphenyl) and fractionation surrogate compounds (2-bromonapthalene and 2-fluorobiphenyl). Prepare stock standard solutions by diluting purchased aromatic hydrocarbon standard (Absolute Standards, part number 51073 at 2000 mg/L or equivalent) and surrogates with methylene chloride to a final concentration of 100 mg/L.

7.2.2 Aliphatic Hydrocarbon Standard: The aliphatic hydrocarbon standard consists of the 14 normal alkanes listed in Table 1, naphthalene, 2-methylnaphthalene, and a surrogate compound (i.e., 1-chloro-octadecane). Prepare stock standard solutions by diluting purchase aliphatic hydrocarbon standard (Absolute Standards, part number 93459 at 2000 mg/L or equivalent) and surrogates with methylene chloride to a final concentration of 100 mg/L.

7.2.3 Stock standard solutions must be replaced after 6 months, or sooner if comparison with check standards indicates a problem.

7.2.4 Calibration standards are prepared by serial dilution of the stock standard as described in Section 9.10 and in Table 2.

7.3 Petroleum Reference Spiking Solution

7.3.1 The petroleum reference spiking solution consists of an API or commercial diesel fuel standard. Prepare stock standard solutions by accurately weighing approximately 0.02500 g of neat product. Dissolve neat product in acetone and dilute to volume in a 10-mL volumetric flask. An appropriately diluted aliquot of the stock solution may be used to evaluate method performance.

7.4 Surrogate Standards

7.4.1 Surrogate standards are used to monitor the efficiency of sample extraction, chromatographic, and calibration systems.

7.4.2 The recommended surrogate standards are 1-chloro-octadecane (COD) and ortho-terphenyl (OTP). These are available as a 2000 mg/L mix from Absolute Standards, part number 51075.

7.4.3 Surrogate Spiking Solution: The recommended surrogate spiking solution is comprised of a mixture of the COD and OTP surrogate standards. Prepare a surrogate spiking solution which contains the surrogate standards at a concentration of 40 mg/L in acetone. Each sample, blank, and matrix spike is fortified with 1.0 mL of the surrogate spiking solution. The use of higher concentrations are permissible and advisable when spiking highly contaminated samples.

7.5 Fractionation Surrogate Standards

7.5.1 The fractionation surrogate standards are added to the sample (hexane) extract just prior to fractionation. The purpose of the fractionation surrogate standards is to monitor the efficiency of the fractionation process, and ensure that unacceptable quantities of naphthalene and substituted naphthalenes are not being eluted into the aliphatic extract.

7.5.2 The recommended fractionation surrogate standard is 2-bromonaphthalene. Other alternative fractionation surrogate compounds, including 2-fluorobiphenyl are permissible, provided that a demonstration is made that such compounds exhibit polarities/fractionation properties similar to naphthalene.

7.5.3 The fractionation surrogate standards are prepared from a purchased certified standard (Absolute Standards, part number 51039 at 2000 mg/L or equivalent).

7.5.4 The fractionation surrogate spiking solution is comprised of 2-bromonaphthalene and 2-fluorobiphenyl (optional) prepared in hexane at concentrations of 20 mg/L. An aliquot of 10 uL of the fractionation surrogate spiking solution is added to the 1 mL EPH sample extract prepared in accordance with the provisions of Sections 9.2 and 9.3. Alternative concentrations/volumes of the fractionation surrogate spiking solution are permissible.

7.6 Matrix Spiking Solution

7.6.1 Analytes from each hydrocarbon group (i.e., aromatic and aliphatic hydrocarbons) are used in a matrix spiking solution, which is prepared using a separate source from the calibration standards. This separate source requirement can be waived if an Initial Calibration Verification (ICV) is analyzed.

7.6.2 The spiking solution, consisting of all normal alkanes in Table 1 and all PAHs in Table 2, is prepared in acetone at a concentration of 50 mg/L (The concentration should be between the mid and upper level of calibration).

7.6.3 The samples selected as the matrix spike are fortified with 1.0 mL of the matrix spiking solution.

Analytical Note: The matrix spiking solution should always be brought to room temperature before use to avoid dissolution of the highest boiling (marginal solubility) hydrocarbon standards.

7.7 Fractionation Check Solution

7.7.1 Prepare a fractionation check solution in hexane containing 100 mg/L of the aliphatic hydrocarbon standard (C_9-C_{36} alkanes) and 100 mg/L of the aromatic hydrocarbon standard (target PAH analytes). The final solution will contain 14 alkanes and 17 PAHs at concentrations of 100 mg/L each. Alternative concentrations are permissible.

7.8 Initial Calibration Verification

7.8.1 Prepare a second source aliphatic hydrocarbon standard (UltraScientific, part number SMA-310-1) and COD surrogate at a final concentration of 50 mg/L

7.8.2 Prepare a second source aromatic hydrocarbon standard (UltraScientific, part number SMA-300-1) and OTP and fractionation surrogate at a final concentration of 50 mg/L.

VIII. Sample Collection, Preservation and Handling

8.1 Aqueous Samples

8.1.1 It is good practice to instruct field personnel to collect aqueous samples in duplicate. Samples must be collected in 1 liter amber glass bottles with Teflon-lined screw caps.

8.1.2 Aqueous samples must be preserved at the time of sampling by the addition of a suitable acid to reduce the pH of the sample to less than 2.0. This may be accomplished by the addition of 5 mL of 1:1 HCl to a 1 liter sample. The uses of alternative acids are permissible. Following collection and addition of acid, the sample must be cooled to 4 ± 2° C.

8.1.3 A chain of custody form must accompany all sample bottles and must document the date and time of sample collection and preservation method used. The laboratory must determine the pH of all water samples as soon as possible after sample receipt and prior to sample extraction. Any sample found to contain a pH above 2 must be so noted on the laboratory/data report sheet and the pH must be adjusted as soon as possible.

8.1.4 Any sample received by the laboratory that is not packed in ice or cooled to 4 ± 2 °C must be so noted on the laboratory/data report sheet. The temperature of the cooler must be recorded by the laboratory upon receipt.

8.1.5 Aqueous samples must be extracted within 14 days of collection, and analyzed within 40 days of extraction.

8.2 Soil/Sediment Samples

8.2.1 Soil and sediment samples are collected in 4 oz. (120 mL) amber wide-mouth glass jars with Teflon-lined screw caps.

8.2.2 Soil and sediment samples must be cooled to 4 ± 2 °C immediately after collection.

8.2.3 A chain of custody form must accompany all sample bottles and must document the date and time of sample collection and preservation method used.

8.2.4 Any sample received by the laboratory that is not packed in ice or cooled to 4 ± 2 °C must be so noted on the laboratory/data report sheet. The temperature of the cooler must be recorded by the laboratory upon receipt.

8.2.5 Soil and sediment samples must be extracted within 14 days of collection, and analyzed within 40 days of extraction.

8.2.6 Alternatively, samples may be frozen (- 10 °C) in the field or in the laboratory. Samples frozen in the laboratory must be preserved at 4 ± 2 °C from the time of sampling and frozen within 48 hours.

8.3 A summary of sample collection, preservation, and holding times is provided in Table 3.

IX. Sample Analysis

9.1 Samples are extracted using methylene chloride and solvent-exchanged into hexane. EPH extraction may be accomplished manually or by automated methods. In this Section a detailed description of manual separatory funnel liquid-liquid extraction for aqueous samples (SW-846 Method 3510) and the Pressurized Solvent extraction procedure (SW-846 Method 3545) for soils and/or sediments are presented to demonstrate general extraction concepts for petroleum products. The applicable SW-846 Method should be consulted for specific details for the other approved EPH extraction procedures

9.2 Water Extraction by Separatory Funnel Liquid-Liquid Extraction

9.2.1 Mark the meniscus on the 1 liter sample bottle (for later volume determination) and transfer the contents to a 2-liter separatory funnel. For blanks and quality control samples, pour 1 liter of reagent water into the separatory funnel. For all samples, blanks, LCSs, LCSDs and matrix spikes add 1.0 mL of the concentrated surrogate spiking solution (see Section 7.4) directly to the separatory funnel. For samples selected for spiking, also add 1.0 mL of the matrix spiking solution.

9.2.2 Check the pH of the sample with wide-range pH paper. Note the pH in the laboratory notebook. The pH of the sample must be adjusted to pH <2.

9.2.3 Add 60 mL methylene chloride to the sample bottle to rinse the inner walls of the container, and then add this solvent to the separatory funnel.

9.2.4 Seal and shake the separatory funnel vigorously for at least three (3) minutes with periodic venting to release excess pressure.

Important: Methylene chloride creates excessive pressure very rapidly; therefore, venting should be done immediately after the separatory funnel has been sealed and shaken once.

9.2.5 Allow the organic layer to separate from the water phase for a minimum of 5 minutes. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Dry the extract by passing it through a glass powder funnel containing anhydrous sodium sulfate or other suitable drying agent. Collect the solvent extract in a Zymark condenser tube.

9.2.6 Repeat the extraction two more times using additional 60 mL portions of solvent. Combine the three solvent extracts, after passing each extract through anhydrous sodium sulfate, in the Zymark condenser tube. (Steps 9.2.3 to 9.2.5)

9.2.7 For sample volume determination add water to the sample bottle to the level of the meniscus previously marked and transfer this water to a graduated cylinder.

9.2.8 Turn on the Turbovap unit along with the nitrogen supply to the unit. Check the level of the water in the water bath. If necessary, add deionized water so as to bring the level to the evaporation tube holder. Set the water bath temperature at 42 °C. When the temperature of the water bath reaches 42 °C, open the cover and place the tubes with extracts in the appropriate positions. Turn on nitrogen flow to the appropriate positions. Close the cover; this starts the concentration process. Rinse down the walls of the tubes with approximately 10 mL of solvent periodically during the evaporation cycle. Adjust the final volume of the extract to 1.0 mL with $MeCl_2$.

9.2.9 Exchange the methylene chloride with hexane by adding 20 mL of hexane to the Zymark condenser tube. Concentrate the extract to 1.0 mL.

9.2.10 Transfer the extract to a labeled 2.5 mL screw cap vial. If a TPH analysis is to be conducted, without fractionation, proceed to Section 9.5.

Analytical Note: Due caution must be exercised during blow down to avoid losses of the more volatile (C_9 through C_{12}) EPH components. The fractionation extract (or any extract) volume should never be reduced below 1 mL in this or any other step to minimize volatilization losses.

9.2.11 Add 10 uL of the concentrated fractionation surrogate (see Section 7.5) spiking solution to the 1 mL hexane extract.

9.2.12 Record the sample preparation information for the extraction and concentration steps. At a minimum, record the date, sample laboratory number, sample volume, volume and

concentration of added surrogates and matrix spike solutions, final extract volume, and any deviations or problems associated with the extraction of the samples.

9.2.13 The 1 mL extract is now ready to be cleaned and fractionated using either commercially-available or self-packed silica gel SPE cartridges. If cleanup will not be performed immediately, transfer the extract to a Teflon-lined screw-cap vial, label, and store at -10 °C.

9.2.14 For cleanup and fractionation, refer to Section 9.4.

9.3 Soil and/or Sediment Extraction using Pressurized Solvent Extraction

9.3.1 Prepare a sample by transferring a 10g aliquot to a tared solvent rinsed glass beaker. Sample particle size reduction must be performed on any sample that will not pass through a 100-200 mesh (150-75um) sieve. Add approximately 30 g of diatomaceous earth (DE) and mix thoroughly until a free flowing homogenous mixture is established. **Samples mus t be dry and free flowing for a proper extraction.** Add 1.0 mL of the surrogate spiking solution (see Section 7.4) to all samples, blanks, LCSs, LCSDs and matrix spikes. Thoroughly mix the surrogate spiking solution into the sample. For samples selected for spiking, add 1.0 mL of the matrix spiking solution. Thoroughly mix the matrix spiking solution(s) into the sample.

9.3.2 Setup a solvent cleaned 60 mL cell with a bottom filter and cap (Dionex logo on the top end). Add additional diatomaceous earth through a funnel to cover the bottom of the cell. Alternately, use 100 mL cells to accommodate the sample volume required. Clean the screw threads, and hand tighten top cap. Repeat procedure for all samples to be analyzed in the batch (up to 20 samples). Load the cells into the appropriate locations in the ASE tray with the Dionex logos on top. Set up the labeled collection bottles according to the manufacturer's recommendation.

9.3.3 Run one to two rinse cycles (priming) prior to analysis if changing over solvent types, refilling the reservoir, or if air bubbles are trapped in the solvent line.

9.3.4 Extract the samples utilizing "Method 3" of the saved ASE methods. See Table 5 for specific method parameters. Allow the method to run and collect the extracts in the collection bottles.

9.3.5 Dry the extract by passing it through a glass powder funnel containing anhydrous sodium sulfate. Collect the dried extract in a Zymark condenser tube. Rinse each collection bottle with 5-10 mL of solvent; add the washings to the appropriate sample extract in the Zymark condenser tube. Concentrate the sample as in Section 9.5.

9.4 Silica Gel Cleanups and Fractionation

9.4.1 The silica gel cleanup and fractionation step is a critical and highly sensitive procedure. Small changes in the volumes of eluting solvents, fractionation equipment, and/or fractionation techniques can significantly impact the proportion of hydrocarbons segregated in either the aliphatic or aromatic fractions. Considerable care and attention is required to ensure satisfactory results.

9.4.2 Each sample fractionation requires 1 mL of sample extract. Re-fractionation would be necessary if problems are experienced during the initial fractionation effort, if

unacceptable breakthrough is noted for naphthalene and 2-methylnaphthalene in the LCS and/or LCSD, and/or if unacceptable recoveries are noted for the fractionation surrogate standard.

9.4.3 Silica gel is a regenerative adsorbent of amorphous silica with weakly acidic properties. It is produced from sodium silicate and sulfuric acid. Silica gel can be used for column chromatography and is used for separating analytes from interfering compounds of a different chemical polarity. Silica gel is also used to separate petroleum distillates into aliphatic and aromatic fractions. A 5g/20 mL solid phase extraction (SPE) silica gel cartridge is commercially available (Waters Corporation, part number WAT036930). Alternatively, the use of self-packed columns of activated silica gel may also be used. The use of activated silica gel for general column chromatographic applications is described in detail SW-846 Method 3630C.

9.4.3.1 To ensure satisfactory fractionation, silica gel/cartridges must not be overloaded. It is recommended that loading be limited to no more than 5 mg total hydrocarbons/gram silica gel; for a 1 mL extract fractionated on a 5 gram silica gel cartridge, this would equate to a hydrocarbon extract loading of no greater than 25,000 µg/mL. It should be noted that overloading the column may result in a premature breakthrough of the C11-C22 aromatic hydrocarbon range. If overloading is encountered, the sample must be re-fractionated at a dilution appropriate for the column's maximum loading capacity.

9.4.3.2 Unsealed silica gel/cartridges must be stored in properly maintained desiccators to avoid inadvertent adsorption of ambient moisture. Silica gel that has been exposed to moisture may perform erratically resulting in poor performance manifested by naphthalene/2-methylnaphthalene and fractionation surrogate breakthrough.

Analytical Note: Air-drying the cartridges may adversely affect silica gel performance and is not advised.

9.4.4 The fractionation check solution described in Section 7.7 must be used to evaluate each new lot of silica gel cartridges to re-establish the optimum volume of hexane elutriate. See Appendix 5, Section 5.0 of the method for optimization specifications. It is not uncommon to encounter inconsistent cartridge weights, mesh sizes and/or variable fractionation performance within the same lot of silica gel cartridges. It may be advisable to perform additional intra-lot fractionation performance checks particularly for larger lot sizes (500) of silica gel cartridges. If concerns exist over the presence of contaminants in the silica gel/cartridge, pre-rinse the column with 30 mL of methylene chloride.

9.4.4.1 Rinse the column with 30 mL of hexane, or 60 mL if pre-rinsed with methylene chloride per Section 9.2.3. Let the hexane flow through the column until the head of the liquid in the column is just above the column frit. Close the stopcock to stop solvent flow. Discard the collected hexane.

9.4.4.2 Load 1.0 mL of the combined sample extract and fractionation surrogate solution onto the column. Open the stopcock, and start collecting elutant immediately in a Zymark condenser tube labeled “aliphatics”.

9.4.4.3 Just prior to exposure of the column frit to the air, elute the column with an additional 19 mL of hexane, so that a total of approximately 20 mL of hexane is passed through the column.

9.4.4.4 It is essential that “plug flow” of the sample extract be achieved through the silica gel cartridge/column. Hexane should be added in 1-2 mL increments or dropwise using a pipet, with additions occurring when the level of solvent drops to the point just prior to exposing the column frit to air. The use of a stopcock is mandatory. Care must be taken to ensure that the silica gel is uniformly packed in the column. The analyst must be cognizant of any channeling, streaking, or changes in the silica gel matrix during fractionation; if any of these occur, the procedure must be repeated with another 1 mL volume of sample extract.

9.4.4.5 The amount of hexane used during fractionation is critical. Excessive hexane - as little as 0.5 mL - can cause significant elution of lighter aromatics into the aliphatic fraction. Insufficient hexane will cause low recoveries of the aliphatic fraction. The volume of the hexane fractionation elutriate should not exceed 20 mL.

9.4.4.6 Following recovery of the aliphatic fraction, elute the column with 20 mL of methylene chloride and collect the eluant in a Zymark concentrator tube. Label this fraction "aromatics".

9.5 Final Sample Extract Concentration

9.5.1 Concentrate each of the extracts to a final volume of 1 mL under a gentle stream of nitrogen in the Turbovap.

Analytical Note: Due caution must be exercised during blow down to avoid losses of the more volatile (C_9 through C_{12}) EPH components. The fractionation extract (or any extract) volume should never be reduced below 1 mL in this or any other step to minimize volatilization losses.

9.5.2 Transfer the final 1 mL extracts from each concentrator tube to labeled two-mL glass auto sampler vials with Teflon-lined rubber crimp caps and store at -10 °C.

9.6 Determination of Percent Moisture

9.6.1 Soil and sediment results must be reported on a dry-weight basis. The wet chemistry department will perform the Total Solids (SM2540-G) analysis and determine the percent moisture that will be used in the required calculations. Refer to the Premier Laboratory Total Solids (% Solids) SOP or the EPH method for more information.

9.7 Analytical Conditions

9.7.1 Recommended analytical conditions are presented in Table 6. A chromatographic column with equivalent chromatographic properties, as described in Section 6.4, or alternative chromatographic conditions may be substituted to improve resolution of extractable petroleum hydrocarbons.

9.8 GC Maintenance

9.8.1 Capillary columns: Clean and deactivate the glass injection port liner or replace with a cleaned and deactivated liner.

9.8.2 Break off the first few inches, up to one foot, of the injection port side of the column.

9.8.3 Bake out the column at the maximum temperature of the temperature program. If these procedures fail to eliminate a column degradation problem, it may be necessary to replace the column.

9.9 Retention Time Windows

9.9.1 Before establishing retention time windows, optimize the GC system's operating conditions. Make three injections of the aromatic hydrocarbon and aliphatic hydrocarbon standard mixtures throughout the course of a 72-hr period. Serial injections over less than a 72-hr period may result in retention time windows that are too restrictive.

9.9.2 Calculate the standard deviation of the three absolute retention times for each individual component in the aromatic hydrocarbon standard, the aliphatic hydrocarbon standard, and all surrogates and internal standards.

9.9.3 The retention time window is defined as plus or minus three times the standard deviation of the absolute retention times for each compound in the aliphatic and aromatic standards. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

9.9.4 In those cases where the standard deviation for a particular standard is close to zero the default value of 0.1 minutes should be used. Alternatively, the laboratory may substitute the standard deviation of a closely eluting structurally similar compound to develop a representative statistically-derived retention time window.

9.9.5 The laboratory must calculate retention time windows for each compound in the aliphatic and aromatic standards on each GC column and whenever a new GC column is installed. These data must be retained by the laboratory.

9.9.6 EPH retention time (RT) windows are defined as beginning 0.1 minutes before the RT of the beginning marker compound and ending 0.1 minutes after the RT of the ending marker compound, except for n-C_{19}, which is both a beginning and ending marker compound for two different ranges.

9.9.7 The C_9 - C_{18} aliphatic hydrocarbon range ends immediately (0.1 min) before the elution of the n-C_{19} peak. The C_{19} - C_{36} aliphatic hydrocarbon range begins 0. 1 min before the elution of the n-C_{19} peak; therefore, there is no overlap of the two ranges and the n-C_{19} peak is only included in the C_{19} - C_{36} aliphatic hydrocarbon range.

9.9.8 EPH marker compounds and windows are summarized below:

EPH Marker Compounds

Hydrocarbon Range	Beginning Marker	Ending Marker
C_9-C_{18} Aliphatic Hydrocarbons	0.1 min before n-Nonane	0.1 min before n-Nonadecane
C_{19}-C_{36} Aliphatic Hydrocarbons	0.1 min before n-Nonadecane	0.1 min after n-Hexatriacontane
C_{11}-C_{22} Aromatic Hydrocarbons	0.1 min before Naphthalene	0.1 min after Benzo(g,h,i)Perylene

9.10 Calibration

9.10.1 The use of calibration factors (CF) is the preferred approach to determine the relationship between the detector response and the analyte and collective range concentrations. It is also permissible to utilize linear regression to calculate the slope and y-intercept that best describes the linear relationship between the analyte and collective range concentrations and the instrument response. The linear regression approach for analytes and collective ranges is described in Appendix A of this SOP.

9.10.2 Prepare aromatic and aliphatic hydrocarbon calibration standards from the stock standard solution at a minimum of five concentrations (i.e., 1x, 10x, 50x, 100x and 200x) by adding volumes of one or more stock standard solutions to 2 mL vials and diluting to 1.0 mL with methylene chloride and hexane, respectively. The surrogate OTP and the fractionation surrogates are included in the aromatic hydrocarbon standard; the surrogate COD is included in the aliphatic hydrocarbon standard. The lowest concentration (1x) determines the minimum working range of the calibration curve and defines the reporting limit (RL) for individual target analytes. The highest concentration (200x) defines the maximum upper working range of the calibration curve. Target analytes may not be reported above this concentration without sample dilution. Reporting limits for collective EPH aliphatic and aromatic hydrocarbon ranges are discussed in Section 12.0. The collective concentrations of individual EPH aliphatic and aromatic hydrocarbon ranges are provided in Table 6. Standard preparation instructions are in Table 2.

Table 6: Recommended Calibration Standard Concentrations
(2 µL injection)

Component	Aliphatic Concentration in mg/L						
	1	**5**	**10**	**20**	**50**	**80**	**100**
C_9-C_{18} aliphatic (6 components)	6	30	60	120	300	480	600
C_{19}-C_{36} aliphatic (8 components)	8	40	80	160	400	640	800
COD	1	5	10	20	50	80	100

Component	PAH Concentration in mg/L							
	0.2*	**2**	**5**	**10**	**20**	**50**	**80**	**100**
C_{11}-C_{22} aromatics (17 component)	3.4	34	85	170	340	850	1360	1700
PAH's	0.2	2	5	10	20	50	80	100
OTP	0.2	2	5	10	20	50	80	100
Fractionation Surrogate	0.2	2	5	10	20	50	80	100

Component	TPH Concentration in mg/L						
	1	**5**	**10**	**20**	**50**	**80**	**100**
TPH C_9-C_{36}	14	70	140	280	700	1120	1400
COD	1	5	10	20	50	80	100

***** The analysis of the 0.2 standard is required to meet the detection limits of several target PAH analytes. It is usually not included in the aromatic C_{11}-C_{22} range. Consult any project specific detection limits to determine if it will be required to be included in this range.

9.10.3 Target PAH Analyte Calibration: Tabulate peak area responses against the concentration injected. The ratio of area response to the concentration injected, defined as the calibration factor (CF), may be calculated for target PAH analytes using Equation 1. The percent relative standard deviation (% RSD) of the calibration factor must be equal to or less than 25% over the working range for the analyte of interest, as determined using Equation 2. When this condition is met, linearity through the origin may be assumed, and the average calibration factor may be used in lieu of a calibration curve.

Equation 1: Calibration Factor (CF) for Target PAH Analytes

$$CF = \frac{\text{area of peak}}{\text{concentration injected (ng/ul)}}$$

Equation 2: Percent Relative Standard Deviation

$$\%\ RSD = \frac{\text{standard deviation of X CFs}}{\text{average of X CFs}} \times 100$$

where: X = the number of calibration levels.

9.10.4 Hydrocarbon Range Calibration: A calibration factor must also be established for each hydrocarbon range of interest. Calculate the CFs for C_9-C_{18} aliphatic hydrocarbons, C_{19}-C_{36} aliphatic hydrocarbons and C_{11}-C_{22} aromatic hydrocarbons from the appropriate FID chromatogram. Tabulate the summation of the peak areas of all components in that fraction (i.e. C_9-C_{18} aliphatic hydrocarbons, 6 components) against the total concentration injected. The results can be used to calculate the ratio of the peak area response summation to the concentration injected, defined as the CF, for the hydrocarbon ranges using Equation 3. The % RSD of the calibration factor must be equal to or less than 25% over the working range for the hydrocarbon range of interest, as determined using Equation 2.

Important: For the calculation of calibration factors (CFs), the area for the surrogates must be subtracted from the area summation of the range in which they elute (e.g., COD is subtracted from the C_{19} - C_{36} aliphatic hydrocarbon range). The areas associated with naphthalene and 2-methylnaphthalene in the aliphatic range standard must be subtracted from the uncorrected collective C_9-C_{18} aliphatic hydrocarbon range area prior to calculating the CF.

Equation 3: Range Calibration Factor: Hydrocarbon Ranges

$$\text{Range CF} = \frac{\text{area summation of range components}}{\text{total concentration injected (ng/uL)}}$$

9.10.5 At a minimum, the calibration factor must be verified on each working day, after every 20 samples or every 24 hours (whichever is more frequent), and at the end of the analytical sequence by the injection of a mid-level continuing calibration standard to verify instrument performance. If the percent difference (% D) for any analyte varies from the predicted response by more than ± 25%, as determined using Equation 4, a new multi-point calibration must be performed for that analyte. Greater percent differences are permissible for n-nonane. If the % D or percent drift for n-nonane is greater than 30, note the nonconformance in the case narrative. It should be noted that the % D values are calculated when CFs are used for the initial calibration and percent drifts are calculated when calibration curves using linear regression are used for the initial calibration (see Section 10.4.3.1 of the method).

Equation 4: Percent Difference (% D)

$$\% D = \frac{CF_{avg} - CF_{cc}}{CF_{avg}} \times 100$$

where: CF_{avg} = average calibration factor calculated from initial calibration.
CF_{cc} = calibration factor calculated from continuing calibration standard.

Equation 5: Percent Drift

$$\% \text{ Drift} = \frac{\text{calculated concentration} - \text{theoretical concentration}}{\text{theoretical concentration}} \times 100$$

9.10.6 TPH Analysis: For TPH analysis without fractionation, calibration factors are developed based upon the response of all 14 aliphatic components using Equation 3.

9.11 GC Analysis

9.11.1 Samples are analyzed in a group referred to as an analytical batch. For methods that require extraction prior to analysis, such as EPH, the number of samples that comprise an analytical batch is generally limited to 20 samples plus the requisite QC samples processed concurrently with the extraction batch. The analytical sequence begins with instrument calibration (initial or continuing) followed by up to 20 samples interspersed with blanks and other QC samples and closed with a mid-range continuing calibration standard. The analytical sequence ends when one or more analytical batches have been processed or when any required qualitative and/or quantitative QC criteria are exceeded.

9.11.2 Aliphatic and aromatic extracts are introduced into the gas chromatograph by direct injection.

9.11.3 Inject 2 µL of the sample extract using the solvent flush technique. Smaller volumes may be injected if automatic devices are employed. Record the volume injected to the nearest 0.05 µL and the resulting peak size in area units. It is required that the sample and calibration standard injection volume be consistent.

9.11.4 Establish daily retention time windows for each analyte of interest. Use the absolute retention time for each analyte as the midpoint of the window for that day. The daily retention time window equals the midpoint ± three times the standard deviation determined in Section 9.9. Alternatively, the default value of 0.1 minutes may be used for the daily retention time window.

9.11.4.1 Identification of a target PAH analyte occurs when a peak from a sample chromatogram falls within the daily retention time window. Confirmation on a second GC column or by GC/MS analysis may be necessary, if warranted by project's data quality objectives.

9.11.4.2 Validation of GC system qualitative performance must be accomplished by the analysis of mid-level standards within the analysis sequence. If

the retention times of the target PAH analytes fall outside their daily retention time window in the standards, the system is out of control. In such cases, the cause of the non-conformance must be identified and corrected.

9.11.5 Aliphatic and aromatic ranges of interest are determined by the collective integration of all peaks that elute between specified range "marker" compounds. Due to the variability in software approaches and applications to collective peak area integration, it is recommended that a manual verification be initially performed to document accurate integration.

9.11.6 When quantifying on a peak area basis by external calibration, collective peak area integration for the fractional ranges, or TPH, must be from baseline (i.e. must include the unresolved complex mixture "hump" areas). For the integration of individual target PAH analytes, surrogate compounds, and internal standards, a valley-to-valley approach should typically be used, though this approach may be modified on a case-by-case basis by an experienced analyst. In any case, the unresolved complex mixture "hump" areas must not be included in the integration of individual target PAH analytes, surrogate compounds, and internal standards.

9.11.7 Baseline correction using a system solvent blank is only permissible for the calculation of aliphatic and aromatic hydrocarbon range concentrations when conducted in accordance with the procedures and requirements specified in Section 11.2.4.

9.11.8 If the target or diesel PAH analytes are to be quantitated using this method, and the response for an individual analyte exceeds the highest calibration concentration, dilute the extract and reanalyze. The samples must be diluted so that all peaks fall within the calibration range of the detector and are bracketed by upper and lower calibration standards.

9.11.9 For non-target analytes eluting in the aliphatic, aromatic or TPH fractions, the upper linear range of the system should be defined by peak height measurement, based upon the maximum peak height documented for an aliphatic or aromatic standard within the fraction that is shown to be within the linear range of the detector.

9.11.10 Analytical conditions that require sample dilution include:

a. The concentration of one or more of the target analytes exceed the concentration of their respective highest calibration standard,
b. Any non-target peak eluting within any aliphatic or aromatic range exceeds twice the peak height documented for the highest range-specific calibration standard, or
c. Anytime a saturated chromatographic peak (flat-topped peak) is encountered.

9.11.11 When sample extracts are diluted, the reporting limit (RL) for each target analyte and/or range must be adjusted (increased) in direct proportion to the dilution factor (DF).

$$\textbf{DF} = \frac{\textbf{sample extract volume - diluent volume}}{\textbf{sample extract volume}}$$

The revised RL for the diluted sample is then calculated as:

RL_d = DF x lowest calibration standard for target PAH analyte (or hydrocarbon range)

9.11.12 It should be understood that samples with elevated RLs as a result of a dilution may not be able to satisfy "MCP program" reporting limits in some cases if the RL_d is greater than the applicable MCP standard or criterion to which the concentration is being compared. Such increases in RLs are the unavoidable but acceptable consequence of sample extract dilution that enable quantification of target analytes which exceed the calibration range. All dilutions must be fully documented in the analytical report.

Analytical Note: Over dilution is an unacceptable laboratory practice. The target post-dilution concentration for the highest concentration target analyte should be at least 60 - 80% (must be at least 50% for MCP samples and 60% for RCP samples) of its highest calibration standard. This will avoid unnecessarily high reporting limits for other target analytes, which did not require dilution.

9.12 Calculations

9.12.1 The concentration of target PAH analytes and hydrocarbon ranges in a sample may be determined by calculating the concentration of the analyte or hydrocarbon range injected, from the peak area response, using the calibration factor determined in Section 9.8.2 and 9.8.3. If linear regression is used for calibration, refer to Appendix A for sample concentration calculations. The Enviroquant program and LIMS will perform all necessary calculations. See Section 11.1 and 11.2 for additional steps needed to ensure that the calculations are performed correctly. These equations are provided for informational purposes to allow the analyst a clear understanding on how the final reported results are calculated.

9.12.2 Aqueous Samples

9.12.2.1 The concentration of a specific analyte or hydrocarbon range in an aqueous sample may be calculated using Equations 6 and 7, respectively.

Equation 6: Target PAH Analytes in Aqueous Samples

$$\textbf{analyte concentration (µg/L)} = \frac{(A_x)(D)(V_t)}{(CF)(V_s)}$$

Equation 7: Hydrocarbon Ranges in Aqueous Samples

$$\textbf{range concentration (µg/L)} = \frac{(A_x)(D)(V_t)}{(\text{Range CF})(V_s)}$$

where: A_x = Response for the analyte, hydrocarbon range, or TPH in the sample. Units must be in area counts for target PAH analytes and must be an area count summation for the hydrocarbon ranges and TPH.

D = Dilution factor*; dimensionless.

CF = Average calibration factor for target PAH analyte, determined in Section 9.10

Range CF = Average calibration factor for hydrocarbon range or TPH, determined in Section 9.10

V_t = Volume of total extract, µL (fractionation + surrogate volume)

V_s = Volume of sample extracted, mL.

9.12.3 Non-aqueous samples

9.12.3.1 The concentration of a specific analyte or hydrocarbon range in a non-aqueous sample may be calculated using Equations 7 and 8, respectively.

Equation 8: Target PAH Analytes in Non-Aqueous Samples

$$\textbf{analyte concentration (µg/kg)} = \frac{(A_x)(D)(V_t)}{(CF)(W_d)}$$

Equation 9: Hydrocarbon Ranges and TPH in Non-Aqueous Samples

$$\textbf{range concentration (µg/L)} = \frac{(A_x)(D)(V_t)}{(\text{range CF})(W_d)}$$

where: W_d = Dry weight of sample, g

A_x, V_t, D, CF, and Range CF have the same definition as described above for Equations 6 and 7.

9.12.4 Calculation of Dry Weight of Sample

9.12.4.1 In order to calculate the dry weight of sample extracted (W_d), it is necessary to determine the moisture content of the soil/sediment sample, using the procedure outlined in Section 9.6. Using the data obtained from Section 9.6, W_d is calculated using Equations 10 through 12.

Equation 10: Percent Moisture

$$\textbf{\% moisture} = \frac{\textbf{grams wet sample} - \textbf{grams dry sample}}{\textbf{grams wet sample}} \times 100$$

Equation 11: Percent Solids

% dry solids = 100 – (% moisture)

Equation 12: Dry Weight of Sample

$$W_d = \frac{\text{(\% dry solids)(grams of extracted sample)}}{100}$$

X. Quality Control

10.1 General Requirements and Recommendations

10.1.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability (IDLC) and an ongoing analysis of spiked samples to evaluate and document the quality of data. The initial demonstration of laboratory capability should be repeated whenever new staff are trained or significant changes in instrumentation or the method (i.e., new extraction method, etc.) are made. The laboratory must maintain records to document data quality. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance standards for the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the analytical system was in-control when the measurements were performed.

10.1.2 A system solvent blank must be run after all highly contaminated samples to minimize the potential for sample carryover. For purposes of this analytical requirement, any sample with an on-column concentration greater than the highest calibration standard is considered "highly contaminated" (see Section 4.4).

10.2 Batch Analytical Quality Control Samples

10.2.1 At a minimum, for each analytical batch (up to 20 samples) or every 24 hours, whichever come first, a beginning and ending continuing calibration standard (CCAL) must be analyzed. For analytical batches with more than 10 samples, the analysis of an additional mid-range CCAL should also be considered. However, it should be noted that the analysis of the CCAL is required prior to sample analysis, after every 20 samples or every 24 hours, whichever comes first, and at the end of an analytical sequence, at a minimum.

10.2.2 At a minimum, for each analytical batch (up to 20 samples of similar matrix), a laboratory method blank, a laboratory control sample (LCS), and a LCS duplicate must also be analyzed and results analyzed as part of the laboratory's continuing quality control program. The blank and quality control samples fortified with known concentrations and volumes of analytical standards should be carried through the complete sample preparation and measurement processes.

10.2.3 It should be noted that field QC samples (field blanks, duplicates, matrix spikes and matrix spike duplicates) are run on pre-identified field samples at the request

of the data user. Coordination with the laboratory is required to assure that adequate sample volume is available.

10.2.4 The recommended analytical sequence is as follows:

a. Analytical batch opening initial calibration (ICAL) or mid-range CCAL [REQUIRED]
b. ICAL verification [REQUIRED for RCP samples and for MCP samples only if separate source standard is not used for LCS/LCSD]
c. LCS sample [REQUIRED]
d. LCSD sample [REQUIRED only for MCP samples]
e. Method Blank [REQUIRED]
f. Up to 20 Samples
g. Matrix Duplicate sample [As requested by data user]
h. Matrix Spike/MS Duplicate [As requested by data user]
i. Optional mid-range CCAL (consider after 10 samples)
j. Closing mid-range CCAL after 20 samples and at end of analytical batch [REQUIRED]

1. May be used as opening CCAL for the next analytical batch if batches are processed continuously.

10.3 Minimum Instrument QC

10.3.1 The instrument must be able to achieve adequate separation and resolution of peaks and analytes of interest.

10.3.1.1 The n-nonane (n-C9) peak must be adequately resolved from the solvent front of the chromatographic run.

10.3.1.2 The surrogates COD and OTP must be adequately resolved from any individual components in the aliphatic hydrocarbon and aromatic hydrocarbon standards.

10.3.1.3 All peaks of interest in the aliphatic hydrocarbon standard must be adequately resolved to baseline. In the aromatic hydrocarbon standard, baseline separation is expected for phenanthrene and anthracene. Benzo(a)anthracene, chrysene, benzo(b)fluoranthene, benzo(k)fluoranthene, dibenzo(a,h)anthracene, and indeno(1,2,3-cd)pyrene are not expected to be chromatographically separated to baseline and may be reported as an unresolved mixture, unless adequate resolution is obtained .

10.3.1.4 For the purposes of this method, adequate resolution is assumed to be achieved if the height of the valley between two peaks is less than 25% of the average height of the two peaks.

10.3.2 Retention time windows must be re-established for target EPH analytes each time a new GC column is installed, and must be verified and/or adjusted on a daily basis. (See Sections 9.9)

10.3.3 Calibration curves, calibration factors, or response factors must be developed based upon the analysis of calibration standards prepared at a minimum of 5 concentration levels. The linearity of calibration or response factors may be assumed if the % RSD over the working range of the curve is less than or equal to 25%. Alternatively, if linear regression analysis is used for quantitation (i.e., calibration curve), the correlation coefficient (r) must be at least 0.99.

10.3.4 In order to demonstrate the absence of aliphatic mass discrimination, the response ratio of C_{28} to C_{20} must be at least 0.85. If <0.85, this nonconformance must be noted in the laboratory case narrative. The chromatograms of CCAL for aromatics must be reviewed to ensure that there are no obvious signs of mass discrimination.

10.3.5 Due care must be exercised to assure that the peaks for naphthalene and n-dodecane in the aliphatic hydrocarbon fraction are adequately resolved to allow for an accurate determination of the naphthalene concentration in the LCS/LCSD pair.

10.4 Ongoing Method QC Demonstrations

10.4.1 Each sample, blank, LCS, LCSD, MS, and MSD must be fortified with the surrogate spiking solution. Required surrogate recovery is 40% to 140%. At a minimum, when surrogate recovery from a sample, blank, or QC sample is less than 40% or more than 140%, check calculations to locate possible errors, check the fortifying solution for degradation, and check for changes in instrument performance. If the cause cannot be determined, re-extract and reanalyze the sample if the recovery of one surrogate is <40% or the recoveries of both surrogates are outside the acceptance limits. Re-extraction and reanalysis are not required if one of the following exceptions applies:

a. Obvious interference is present on the chromatogram (e.g., unresolved complex mixture); and

b. If the surrogate exhibits high recovery and associated target analytes or hydrocarbon ranges are not detected in sample.

10.4.2 Analysis of the sample on dilution may diminish matrix-related surrogate recovery problems. This approach can be used as long as the reporting limits to evaluate applicable MCP standards can still be achieved with the dilution. If not, reanalysis without dilution must be performed.

10.4.3 Each sample (field and QC sample) must be evaluated for potential breakthrough on a sample-specific basis by evaluating the % recovery of the fractionation surrogate (2-bromonaphthalene) and on a batch basis by quantifying naphthalene and 2-methylnaphthalene in both the aliphatic and aromatic fractions of the LCS and LCSD. If either the concentration of naphthalene or 2-methylnaphthalene in the aliphatic fraction exceeds 5% of the total concentration for naphthalene or 2-methylnaphthalene in the LCS or LCSD, fractionation must be repeated. If the

fractionation surrogate recovery is outside the 40 – 140% limits, then fractionation must be repeated for the affected sample. NOTE: The total concentration of naphthalene or 2-methylnaphthalene in the LCS/LCSD pair includes the summation of the concentration detected in the aliphatic fraction and the concentration detected in the aromatic fraction.

Analytical Note: Due care must be exercised to assure that the peaks for naphthalene and n-dodecane in the aliphatic hydrocarbon fraction are adequately resolved to allow for an accurate determination of the naphthalene concentration in the LCS/LCSD pair.

10.4.4 At a minimum, with every batch of 20 samples or less the laboratory must extract and analyze the following quality control samples:

10.4.4.1 Continuing Calibration Standard - A mid-range continuing calibration standard, prepared from the same stock standard solution used to develop the calibration curve, must be analyzed prior to sample analysis to verify the calibration state of the instrument. For large analytical batches that contain more than 10 samples, the analysis of an additional mid-range continuing calibration standard is recommended after the analysis of the tenth sample. However, it should be noted that a mid-range continuing calibration standard is required after every 20 samples or every 24 hours (whichever comes first) and at the end of the analytical sequence. If the percent difference or percent drift of any analyte within the continuing calibration standard varies from the predicted response by more than 25%, a new five-point calibration must be performed for that analyte. Greater differences are permissible for n-nonane. If the percent difference or percent drift is greater than 30% for n-nonane, note the nonconformance in the narrative. For the closing continuing calibration standard (analyzed after every 20 samples, every 24 hours, or at end of analytical sequence), four compounds may exhibit percent differences or percent drifts greater than 25% but less than 40%.

10.4.4.2 Laboratory Method Blank - A water or soil laboratory method blank is prepared by fortifying a reagent water or clean sand blank (optional) with 1.0 mL of the surrogate spiking solution. Peaks must not be detected above the reporting limit within the retention time window of any analyte of interest. The hydrocarbon ranges must not be detected at a concentration greater than 10% of the most stringent MCP or RCP cleanup standard. Peaks detected within the retention time window of any analyte or range of interest above a reporting limit must be noted on the data report form. Re-extraction of all associated samples may be warranted.

10.4.4.3 Laboratory Control Sample - A laboratory control sample is prepared by fortifying a reagent water or diatomaceous earth blank with 1.0 mL of the matrix spiking solution. The spike recovery must be between 40% and 140%. Lower recoveries of n-nonane are permissible. If the

recovery of n-nonane is <30%, note the nonconformance in the narrative. Re-extraction of all associated samples is required if criteria are not met.

10.4.4.4 LCS Duplicate – A laboratory control sample duplicate is prepared by fortifying a reagent water or diatomaceous earth blank with 1.0 mL of the matrix spiking solution (see Section 7.6 and Tables 1 and 2). The LCS duplicate is separately prepared, processed and analyzed in the same manner as the LCS and is used as the data quality indicator of precision. The analytical batch precision is determined from the relative percent difference (RPD) of the concentrations (not recoveries) of LCS/LCSD pair. The RPD for individual target PAH analytes and aliphatic and aromatic hydrocarbon range concentrations (sum of the individual aliphatic or aromatic compounds within the specified range) must be = 25.

10.4.4.5 Initial Calibration Verification – An initial calibration verification standard, prepared from a separate source standard than used for initial and continuing calibrations, must be analyzed prior to sample analysis if a separate source standard is not used for the LCS. The recoveries of all target analytes must be between 80-120%. A new five-point calibration must be performed if criteria are not met.

10.4.4.6 System Solvent Blank - If baseline correction will be employed, as specified in Section 11.2.4, a system solvent blank, air blank, and/or system run must be undertaken with every batch, and after the analysis of a sample that is suspected to be highly contaminated. In no case shall baseline correction be used if the instrument baseline drift is more than 25% greater than the average level established by these charts.

10.4.4.7 Fractionation Check Standard – A fractionation check solution is prepared containing 14 alkanes and 17 PAHs at a nominal concentration of 100 mg/L of each constituent. The fractionation check solution must be used to evaluate the fractionation efficiency of each new lot of silica gel / cartridges as described in Appendix 5, Section 5.0 of the method, and establish the optimum hexane volume required to efficiently elute aliphatic hydrocarbons while not allowing significant aromatic hydrocarbon breakthrough. For each analyte contained in the fractionation check solution, excluding n-nonane, the percent recovery must be between 40 and 140%. A 30% recovery is acceptable for n-nonane.

10.4.5 At the request of the data user, and in consideration of sample matrices and data quality objectives, matrix spikes and matrix duplicates may be analyzed with every batch of 20 samples or less per matrix.

10.4.5.1 Matrix duplicates - Matrix duplicates are prepared by analyzing one sample in duplicate. The purpose of the matrix duplicates is to determine the homogeneity of the sample matrix as well as analytical

precision. The RPD of detected results in the matrix duplicate samples must not exceed 50 when the results are greater than 5x the reporting limit.

10.4.5.2 Matrix Spike/Matrix Spike Duplicate - The water or soil MS is prepared by fortifying an actual water or soil sample with 1.0 mL of the matrix spiking solution. The desired spiking level is 50% of the highest calibration standard. However, the total concentration in the MS (including the MS and native concentration in the unspiked sample) should not exceed 75% of the highest calibration standard in order for a proper evaluation to be performed. The purpose of the matrix spike is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate unspiked aliquot and the measured values in the matrix spike corrected for background concentrations. The corrected concentrations of each analyte within the matrix spiking solution must be within 40 - 140% of the true value. Lower recoveries of n-nonane are permissible, but must be noted in the narrative if <30%.

10.4.6 If any of the performance standards specified in this section are not met, the cause of the non-conformance must be identified and corrected before any additional samples may be analyzed. Any samples run between the last QC samples that met the criteria and those that are fallen out must be re-extracted and/or re-analyzed. These QC samples include the opening continuing calibration standard, laboratory method blank, LCS, LCSD, and closing continuing calibration standard. If this is not possible, that data must be reported as suspect.

XI. Data Production and Reporting

11.1 Calibration – using the calibration procedure from Section 9.10, calibrate the GC as follows:

11.1.1 Calculate a CF or linear regression (LR) for each target PAH analyte that comprises the aromatic hydrocarbon standard. This step is not necessary if the target or diesel PAH analytes will not be individually identified and quantitated by the EPH method (i.e., if unadjusted values only will be reported for the hydrocarbon ranges or TPH or if reporting concentrations of target PAH analytes via another method).

11.1.2 Calculate a CF for the surrogates OTP, COD and the fractionation surrogates.

11.1.3 Calculate a collective CF or LR for the total concentration of the C_9-C_{18} aliphatic hydrocarbons. Tabulate the summation of the peak areas of all component standards in that fraction (e.g., C_9-C_{18} aliphatics, 6 components) against the total concentration injected. Do not include any area contribution of the internal standard, naphthalene, and 2-methylnaphthalene. Manually enter this sum in the appropriate initial calibration field in Enviroquant for the applicable calibration level. Do this for each calibration level.

11.1.4 Calculate a CF or LR for naphthalene and 2-methylnaphthalene from the aliphatic hydrocarbon standard. Not required if the same instrument is calibrated, separately, for all aliphatic and aromatic compounds using the same internal standard and resolution of naphthalene from n-C_{12} is demonstrated.

11.1.5 Calculate a collective CF or LR for the total concentration of the C_{19}-C_{36} aliphatic hydrocarbons. Tabulate the summation of the peak areas of all component standards in that fraction (e.g., C_{19}-C_{36} aliphatics, 8 components) against the total concentration injected. Do not include the surrogate COD. Manually enter this sum in the appropriate initial calibration field in Enviroquant for the applicable calibration level. Do this for each calibration level.

11.1.6 Calculate a collective CF or LR for the total concentration of the C_{11}-C_{22} aromatic hydrocarbons. Tabulate the summation of the peak areas of all component standards in that fraction (e.g., C_{11}-C_{22} aromatics, 17 components) against the total concentration injected. Do not include the surrogate OTP, 2-bromonaphthalene, or 2-fluorobiphenyl. Manually enter this sum in the appropriate initial calibration field in Enviroquant for the applicable calibration level. Do this for each calibration level.

11.1.7 For TPH analyses, without fractionation, calculate a collective CF or LR. Tabulate the summation of the peak areas of all component standards in the aliphatic fraction (i.e., 14 components) against the total concentration injected. Do not include surrogates or naphthalene and 2-methylnaphthalene in the aliphatic hydrocarbon standard. Manually enter this sum in the initial calibration field in Enviroquant for the applicable calibration level. Do this for each calibration level.

11.2 Sample Analysis

11.2.1 Aliphatic Fraction

11.2.1.1 Determine the total area count for all peaks eluting 0.1 minutes before the retention time (RT) for C_9 and 0.01 minutes before the RT for C_{19}. It is not necessary to identify or quantitate individual aliphatic compounds within this range.

11.2.1.2 Determine the total area count for all peaks eluting 0.01 minutes before the RT for C_{19} and 0.1 minutes after the RT for C_{36}. It is not necessary to identify or quantitate individual aliphatic compounds within this range.

11.2.1.3 Determine the peak area count for the surrogate standard (COD). Subtract this value from the collective area count value within the C_{19} through C_{36} aliphatic hydrocarbon range.

11.2.1.4 Using the equations contained in Section 9.10, calculate the collective concentrations of C_9 through C_{18} aliphatic hydrocarbons, C_{19} through C_{36} aliphatic hydrocarbons, and the individual concentration of the surrogate COD. The Enviroquant GC software and laboratory LIMS system will perform all necessary calculations, with the exception of any

range in which surrogate or other components must be subtracted out. Use the corrected area count from 11.2.1.3 and divide by the average CF for the C_{19}-C_{36} range. Divide again by 1000 and this will be the adjusted concentration in mg/L. For calculations when using linear regression analysis, see Equations 4.3 and 4.4. See Table 7 for examples of concentration calculations.

Equation 13: Calculation of Concentration using Linear Regression for Target EPH Analytes and Ranges in Aqueous Samples

$$\text{concentration } (\mu g/L) = \left(\frac{A_x - b}{a} \right) \times D$$

where: A_x = response for the analyte or hydrocarbon range in the sample. Units are in area counts for target EPH analytes and the hydrocarbon ranges.

D = dilution factor; if no dilution was made, D = 1, dimensionless

a = slope of the line for target EPH analyte or hydrocarbon range

b = intercept of the line for target EPH analyte or hydrocarbon range

Important: Do not include the area of any surrogate standard in A_x when calculating a range concentration.

11.2.1.5 The concentration of a specific target EPH analyte or hydrocarbon range in a soil or sediment sample may be calculated using linear regression analysis by applying Equation 14.

Equation 14: Calculation of Concentration using Linear Regression for Target EPH analytes and Ranges in Non-Aqueous Samples

$$\text{concentration (ug/kg)} = \left(\frac{A_x - b}{a} \right) \times \frac{(V_t)(D)(V_w)}{(V_i)(W_d)}$$

where: W_d = dry weight of sample, g (see Section 9.10.3)

A_x, a, b, and D have the same definition as for aqueous samples in Equation 13

Important: Do not include the area of any surrogate standard in A_x when calculating a range concentration.

11.2.2 Aromatic Fraction

11.2.2.1 Determine the total area count for all peaks eluting 0.1 minutes before the retention time (RT) for naphthalene and 0.1 minutes after the RT for benzo(g,h,i)perylene.

11.2.2.2 Determine the peak area count for the sample surrogate (OTP) and fractionation surrogate(s). Subtract the total area count from the unadjusted C_{11} through C_{22} aromatic hydrocarbons.

11.2.2.3 Determine the peak area count for the target or diesel PAH analytes.

11.2.2.4 By definition, the collective concentration of the aromatic fraction (and/or TPH) excludes the individual concentrations of the target PAH analytes. Accordingly, a data adjustment step is necessary to adjust the collective range concentration to eliminate "double counting" of analytes.

11.2.2.5 Using the equations contained in Section 9.9, calculate the concentrations of unadjusted C_{11} through C_{22} aromatic hydrocarbons, the surrogate standard (OTP), fractionation surrogate standard(s) and the target or diesel PAH analytes.

11.2.2.6 Subtract the individual concentrations of the target or diesel PAH analytes from the collective concentration of unadjusted C_{11} through C_{22} aromatic hydrocarbons. Only subtract the concentrations of the target or diesel PAH analytes if they are above the reporting limit. It should be noted that the reported target PAH analyte results must be the results used to adjust the C_{11}-C_{22} aromatics results.

11.2.3 Total Petroleum Hydrocarbons

11.2.3.1 Determine the total area count for all peaks eluting 0.1 minutes before the retention time (RT) for C_9 and 0.1 minutes after the RT for C_{36}. It is not necessary to identify or quantitate individual aliphatic compounds within this range.

11.2.3.2 Determine the peak area count for the surrogates used. Subtract these values from the collective area count value.

11.2.3.3 Using the equations contained in Section 9.9, calculate the concentration of unadjusted TPH.

11.2.3.4 If the concentrations of the target or diesel PAH analytes are present above the reporting limit and/or the unadjusted TPH value is above 100 mg/L, the sample must be fractionated. Do not report a value for TPH.

11.2.3.5 For purposes of compliance with the reporting and cleanup standards specified in the Massachusetts Contingency Plan, the concentration of unadjusted C_{11} through C_{22} aromatic hydrocarbons and/or unadjusted TPH may be conservatively deemed to be equivalent to the concentration of C_{11} through C_{22} aromatic hydrocarbons and/or TPH.

11.2.4 Baseline Correction for Instrument Noise Level

11.2.4.1 EPH aliphatic and aromatic hydrocarbon range area data determined by the collective integration of all eluting peaks between the specified EPH range marker compounds (see Table 5) may be corrected by the manual or automatic subtraction of the baseline established by the injection of a system solvent blank. Correction in this manner is not recommended or preferred, but is permissible in cases where all reasonable steps have been taken to eliminate or minimize excessive baseline bias associated with analytical system noise.

11.2.4.2 The instrument baseline must be established by the direct injection of a system solvent blank. The injection of an air blank or activation of a temperature programmed chromatographic run without the injection of any material should be used to verify that the system noise is not attributable to solvent contamination. All system operational elements and parameters must be identical to those of a typical sample run.

11.2.4.3 If baseline correction is used, the baseline must be re-established for every analytical batch by the analysis of a system solvent blank. Baseline correction for EPH aliphatic and aromatic hydrocarbon area data may not be used for any sample for which the area count associated with the baseline correction is greater than 10% of the uncorrected area count for the sample's corresponding collective range.

11.2.5 Contamination of SPE Cartridges

11.2.5.1 Range integration areas may be affected by peaks identified during the injection of a laboratory method blank, and determined to be attributable to the leaching of plasticizers or other contaminants from silica gel SPE cartridges. In general, this contamination affects the C_{11}-C_{22} Aromatics. **Blank correction is not permissible.**

11.2.5.2 The laboratory must report the presence of this contamination in the associated range. Optionally, the laboratory may perform GC/MS analysis of the laboratory method blank extract to demonstrate that the contaminant in question is not a C_{11}-C_{22} aromatic compound. Analysis of only the method blank is acceptable as long as the associated samples exhibit the same contaminant peak at the same retention time. If demonstrated not to be a C_{11}-C_{22} aromatic compound, the contaminant does not need to be included in the calculation of the hydrocarbon range concentration. The laboratory must provide a discussion in the case narrative if this approach is used.

XII. Reporting Limits

12.1 The Reporting Limits (RLs) for Target PAH Analytes shall be based upon the concentration of the lowest calibration standard for the analyte of interest.

12.1.2 The RL must be greater than or equal to the concentration of the lowest calibration standard. Target PAH analytes with calculated concentrations below the RL should be reported as < the specific target analyte's RL (i.e., < 2.0 ug/L).

12.1.3 For GC/MS analysis only, calculated concentrations of target PAH analytes below the RL (lowest calibration standard) may be reported as a "J Value", or equivalent.

12.2 The RLs for hydrocarbon ranges shall be based upon the concentration of the lowest calibration standard for an individual analyte within the range of interest.

12.2.1 The range RL will be set at 100x the concentration of the lowest calibration standard for the associated analyte.

12.2.2 Calculated collective concentrations for EPH aliphatic and aromatic hydrocarbon ranges below the RL should be reported as < range RL (i.e., < 100 ug/L).

12.3 Based on the on-column concentration of 1 ?g/µL for the lowest calibration standard for all analytes, the following reporting limits would be generated for the hydrocarbon ranges:

12.3.1 Aqueous Samples: EPH hydrocarbon range reporting limits would be equivalent to 100 µg/L based on the extraction of 1 liter of sample, a final fractionation extract volume of 2 mL, and a sample injection volume of 1 µL.

12.3.2 Soil/Sediment Samples: EPH hydrocarbon range reporting limits would be equivalent to 20 mg/kg (dry weight basis) based on the extraction of 10 grams of soil, a final fractionation extract volume of 2 mL, and a sample injection volume of 1 µL

Table 1: Aromatic Hydrocarbon Standard and Aliphatic Hydrocarbon Standard

PAH Compound	Retention Time (min.)[1]	Carbon Number	Compound	Retention Time (min.)[1]
Naphthalene	9.11	9	n-Nonane	4.12
2-Methylnaphthalene	11.03	10	n-Decane	5.86
Acenaphthylene	13.46	12	n-Dodecane	9.48
Acenaphthene	13.99	14	n-Tetradecane	12.81
Fluorene	15.46	16	n-Hexadecane	15.80
Phenanthrene	18.13	18	n-Octadecane	18.50
Anthracene	18.29	19	n-Nonadecane	19.76
Ortho-Terphenyl (surrogate)	19.46	20	n-Eicosane	20.96
Fluoranthene	21.52	1-Chloro-octadecane	Surrogate	21.92
Pyrene	22.11	22	n-Docosane	23.20
Benzo(a)Anthracene	25.61	24	n-Tetracosane	25.27
Chrysene	25.73	26	n-Hexacosane	27.19
Benzo(b)Fluoranthene	28.61	28	n-Octacosane	28.97
Benzo(k)Fluoranthene	28.61	30	n-Triacontane	30.63
Benzo(a)Pyrene	29.28	36	n-Hexatriacontane	36.36
Indeno(1,2,3-cd)Pyrene[2]	31.84			
Dibenzo(a,h)Anthracene[2]	31.93			
Benzo(g,h,i)Perylene	32.34			

[1] Results obtained using the column and chromatographic conditions described in Sections 6.4 and 9.5.

[2] Indeno(1,2,3-cd)Pyrene, Benzo(b)Fluoranthene, Benzo(k)Fluoranthene and Dibenzo(a,h)Anthracene may co-elute under the column and chromatographic conditions described in Sections 6.4 and 9.5.

Table 2: Preparation of Stock Standard and Spiking Solutions

Component	Concentration	Aliquot	Final Volume	Final Concentration
PAH Stock			**5.0 mL**	**100 mg/L**
Aromatic Hydrocarbon Std.	2000 mg/L	250 uL		
Fractionation Surrogate	2000 mg/L	250 uL		
OTP Surrogate	1000 mg/L	500 uL		
Aliphatic Stock			**5.0 mL**	**100 mg/L**
Aliphatic Hydrocarbon Std.	2000 mg/L	250 uL		
1-COD Surrogate	10,000 mg/L	50 uL		
EPH Matrix Spike			**25 mL**	**50 mg/L**
Aromatic Hydrocarbon Std.	2000 mg/L	625 uL		
Aliphatic Hydrocarbon Std.	2000 mg/L	625 uL		
EPH Surrogate			**50 mL**	**40 mg/L**
1-COD + OTP	2000 mg/L	1.0 mL		

Table 3: Holding Times and Preservatives for EPH Samples

Matrix	Container	Preservation	Holding Time
Aqueous Samples	1-Liter amber glass bottle with Teflon-lined screw cap	Add 5 mL of 1:1 HCl; Cool to 4 ± 2° C	Samples must be extracted within 14 days and extracts analyzed within 40 days
Soil/Sediment Samples	4-oz. (120 mL) wide-mouth amber glass jar with Teflon-lined screw cap	Cool to 4 ± 2° C	Samples must be extracted within 14 days and extracts analyzed within 40 days of extraction
	4-oz. (120 mL) wide-mouth amber glass jar with Teflon-lined screw cap. Jar should be filled to only 2/3 capacity to avoid breakage if expansion occurs during freezing	Freeze at - 10°C in the field or in the laboratory*.	Samples must be extracted within 14 days of the date thawed and extracts analyzed within 40 days of extraction.

* Samples processed in the laboratory must be preserved at 4 ± 2° C and frozen within 48 hours of the time of collection. Frozen samples may be held for up to one year prior to analysis and must be extracted within 24 hours of thawing.

NOTE: For optimum performance, the sample volumes/weights, solvent volumes, and final extract volumes cited in Sections 9.1.1 and 9.1.2 are recommended. Alternate volumes can be used as long as comparable reporting limits are achieved.

The complete list of approved EPH extraction procedures for water and soil/sediment samples is presented in Table 4. Alternative extraction procedures other than those listed are acceptable, provided that the laboratory can document acceptable matrix - and petroleum product-specific performance. However, use of an alternative extraction procedure is considered a "significant modification"

of the EPH method pursuant to Section 11.3.1.1 and as such would preclude obtaining "presumptive certainty" status for any analytical data produced using an alternative EPH extraction procedure

Table 4 - Approved EPH Extraction Methods		
SW-846 Method	**Matrix**	**Description**
3510C	Aqueous	Separatory Funnel liquid-Liquid Extraction
3520C	Aqueous	Continuous Liquid-Liquid Extraction
3511	Aqueous	Organic Compounds in Water by Microextraction
3540C	Soil/Sediment	Soxhlet Extraction
3541	Soil/Sediment	Automated Soxhlet Extraction
3545A	Soil/Sediment	Pressurized Fluid Extraction (PFE)
3546	Soil/Sediment	Microwave Extraction
3570	Soil/Sediment	Microscale Solvent Extraction (MSE)
3550C	Contaminated Solids [1]	Ultrasonic Extraction
3580A	NAPL	Solvent Dilution

[1] Sonication may only be used for the extraction of highly contaminated (free product) non-soil/sediments (debris). Any other use of ultrasonic extraction is considered a "significant modification" of the EPH Method.

Table 5: ASE Method Values	
Parameter	Method 3
Pressure (psi)	1500
Temp (C)	100
Heat (Min)	5
Static (Min)	5
Flush % (vol)	60
Purge (sec)	60
Cycles	1
Time (min/cell)	15

Table 6: Recommended GC Conditions	
Chromatographic Column	30 m x 0.32 mm I.D., 0.25 µm Restek RTX-5MS
Oven Temperature Program	Initial oven temperature 40°C, hold time 1 min; to 290 °C @ 8°C/min, hold time 7 min
Total Run Time	39.25 min
Sample/auto sampler Injection	2 uL
Gas Flow Rates	Carrier gas - Helium @ 2 to 3 mL/ min
	Oxidizer - Air @ 350 mL/min
	Fuel – Hydrogen @ 65 mL/min
	Make up – Air @ 32.0 mL/min
Injection Port Temperature	285°C
Column Inlet Pressure	20 p.s.i.g.
Detector Temperature	315°C (FID)
Linear Velocity	65.7 cm/sec

Appendix A
Example Concentration Calculations

I. Aliphatics

System Monitoring Compounds		RT	Response	Concentration, mg/L
1) S	Chloro-octadecane	22.18	785043	28.028
Target Compounds		**RT**	**Response**	**Concentration, mg/L**
16) T	C_9-C_{18} Aliphatics	13.03	5967245	150.716
17) T	C_{19}-C_{36} Aliphatics	22.18	4879395	103.337

No adjustment is needed for C_9-C_{18} Aliphatic range because no surrogate elutes in this range.

For the C_{19}-C_{36} Aliphatic range

Subtract surrogate response from applicable range response (C_{19}-C_{36}):

4879395 - 785043 = 4094352

Divide the adjusted area count by the Average RF for the C_{19}-C_{36} range from the initial calibration or continuing calibration:

4094352 ÷ 47.218 ÷ 1000 = 86.712 mg/L adjusted concentration of C_{19}-C_{36}

For the C_{11}-C_{22} Aromatics range

System Monitoring Compounds		RT	Response	Concentration, mg/L
1) S	2-Fluorobiphenyl	12.40	446381	16.108
2) S	2-Bromonaphthalene	14.09	328559	16.809
3) S	Ortho-Terphenyl	19.71	794222	25.892
Target Compounds		**RT**	**Response**	**Concentration, mg/L**
9) T	Phenanthrene	18.37	564197	18.593
11) T	Fluoranthene	21.78	941748	30.347
20) x	C_{11}-C_{22} Aromatics	21.78	40378591	1189.528

Adjusted C_{11}-C_{22} Aromatics Range in mg/L = Total C_{11}-C_{22} Range response – total surrogates response ÷ Avg. RF ÷ 1000 – Total Concentration Target Analytes

Adjusted C_{11}-C_{22} Aromatics Range = 40378591 – (446381+328559+794222) ÷ 33.43 ÷ 1000 – (18.593+30.347) = 1094.363 mg/L

For Linear calibrations

The equation for calculating concentration from a linear calibration is:

$$y = mx + b$$

where: y = compound or range adjusted response
m = slope of the line
b = intercept of the line
x = concentration (unknown)

The slope and intercept can be found in the Enviroquant GC program under Calibration ? Edit Compounds. Go into the compound you are determining concentration for and select "Plot". The Plot will give you the values for the slope and intercept that are being used to calculate concentration. Verify that only initial calibration data is present in the table; delete out any continuing calibration (CC) data. Continuing calibration data is not used in determining concentration.

Subtract surrogate area count from total area count to determine adjusted area count. Use this value for y.

Solve for x by the following equation:

$$x = \frac{(y-b)}{m}$$

Example: C_{19}-C_{36} Aliphatic range has a linear curve fit.

System Monitoring Compounds		RT	Response	Concentration, mg/L
1) S	Chloro-octadecane	22.18	785043	28.028
Target Compounds		**RT**	**Response**	**Concentration, mg/L**
16) T	C_9-C_{18} Aliphatics	13.03	5967245	150.716
17) T	C_{19}-C_{36} Aliphatics	22.18	4879395	103.337

No adjustment needed for C_9-C_{18} Aliphatic range because no surrogate elutes in this range.

For the C_{19}-C_{36} Aliphatic range

Subtract surrogate response from applicable range response (C_{19}-C_{36}):

4879395 – 785043 = 4094352
b = 657000 (value of intercept from plot)
a = 41200 (value of slope from plot)

Subtract the intercept from the adjusted area count; then divide by the slope of the line for the C_{19}-C_{36} range from the initial calibration:

$$\mathbf{x = \frac{(4879395 - 657000)}{41200} = 102.48\ mg/L\ adjusted\ concentration\ of\ C_{19}\text{-}C_{36}}$$

Example Naphthalene* % Breakthrough Calculation	
Naphthalene in Aromatic Fraction (N_{ar})	48 µg/L
Naphthalene in Aliphatic Fraction (N_{al})	1.5 µg/L
Total Naphthalene Concentration (NT_r)	49.5 µg/L

$$\% \text{ Naphthalene Breakthrough} = \frac{N_{al}}{NT_r} \times 100$$

$$\% \text{ Naphthalene Breakthrough} = \frac{1.5}{49.5} \times 100$$

% Naphthalene Breakthrough = 3.0

* may be applied to 2-methylnaphthalene breakthrough calculation also

Date	Previous Revision No.	New Revision No.	Description of Changes	Effective Date	Initiated by
2/29/12	1.0	1.1	Added revision history table Changed format Updated to reflect new CAM requirements	2/29/12	GP, LM

Volatile Organics by GC/MS
Method 8260B

Prepared by: Melisa Montgomery, Quality Assurance Officer
Approved by: Robert Stevenson, Laboratory Director
Reviewed and Implemented by: Ronald Warila, General Manager

Reference

Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition, December 1996, Method 8260B, Revision 2

I. Scope and Application

1.1 Analytes: See Table 1

1.2 Matrices: All liquids, sludges, particulate solids

1.3 Regulations: RCRA and equivalent state regulations

II. Important Notes

2.1 Every analyst performing this procedure must be familiar with the requirements of the Quality Control and Corrective Action section. For convenience, quality control information required to assess data is referenced in the Procedure section. However, if the criteria referenced are not met, the Quality Control and Corrective Action section must be consulted for additional information and appropriate actions to be taken.

2.2 Impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks. The use of non-PTFE tubing, non-PTFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

2.3 The estimated quantitation limit (EQL) of Method 8260 for an individual compound is somewhat instrument dependent and also dependent on the choice of sample preparation/introduction method. Using standard quadrapole instrumentation and the purge-and-trap technique, limits should be approximately 5 µg/kg (wet weight) for soil/sediment samples, 0.5 mg/kg (wet weight) for wastes, and 5 µg/L for ground water (see Table 3).

Somewhat lower limits may be achieved using an ion trap mass spectrometer or other instrumentation of improved design. No matter which instrument is used, EQLs will be proportionately higher for sample extracts and samples that require dilution or when a reduced sample size is used to avoid saturation of the detector.

2.4 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatograph/mass spectrometers, and skilled in the interpretation of mass spectra and their use as a quantitative tool. Note: The laboratory where volatile analysis is performed should be completely free of solvents.

III. Summary of Method

3.1 The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method or by other methods. The analytes are introduced directly to a wide-bore capillary column before being flash evaporated to a narrow-bore capillary for analysis. The column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) interfaced to the gas chromatograph (GC).

3.2 Analytes eluted from the capillary column are introduced into the mass spectrometer via a jet separator or a direct connection. (Wide-bore capillary columns normally require a jet separator, whereas narrow-bore capillary columns may be directly interfaced to the ion source). Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a five-point calibration curve.

3.3 The method includes specific calibration and quality control steps that supersede the general requirements provided in Method 8000.

IV. Interferences

4.1 Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components should be avoided, since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of calibration and reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should change the purge gas source and regenerate the molecular sieve-purge gas filter. Subtracting blank values from sample results is not permitted. If reporting values without correcting for the blank results in what the laboratory feels is a false positive result for a sample, the laboratory should fully explained this in text accompanying the uncorrected data.

4.2 Contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. A technique to prevent this problem is to rinse the purging apparatus and

sample syringes with two portions of organic-free reagent water between samples. After the analysis of a sample containing high concentrations of volatile organic compounds, one or more blanks should be analyzed to check for cross-contamination. Alternatively, if the sample immediately following the high concentration sample does not contain the volatile organic compounds present in the high level sample, freedom from contamination has been established.

4.3 For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds, or high concentrations of compounds being determined, it may be necessary to wash the purging device with a soap solution, rinse it with organic-free reagent water, and then dry the purging device in an oven at 105°C. In extreme situations, the entire purge-and-trap device may require dismantling and cleaning. Screening of the samples prior to purge-and-trap GC/MS analysis is highly recommended to prevent contamination of the system. This is especially true for soil and waste samples. Screening may be accomplished by Method 3820 (Hexadecane Extraction and Screening of Purgeable Organics).

4.4 Many analytes exhibit low purging efficiencies from a 25-mL sample. This often results in significant amounts of these analytes remaining in the sample purge vessel after analysis. After removal of the sample aliquot that was purged, and rinsing the purge vessel three times with organic-free water, the empty vessel should be subjected to a heated purge cycle prior to the analysis of another sample in the same purge vessel. This will reduce sample-to-sample carryover.

4.5 Special precautions must be taken to analyze for methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride. Otherwise, random background levels will result. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing should be constructed from stainless steel or copper tubing. Laboratory clothing worn by the analyst should be clean, since clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination.

4.6 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample container into the sample during shipment and storage. A trip blank prepared from organic-free reagent water and carried through the sampling, handling, and storage protocols can serve as a check on such contamination.

4.7 Use of sensitive mass spectrometers to achieve lower detection level will increase the potential to detect laboratory contaminants as interferences.

4.8 Direct Injection - Some contamination may be eliminated by baking out the column between analyses. Changing the injector liner will reduce the potential for cross-contamination. A portion of the analytical column may need to be removed in the case of extreme contamination. The use of direct injection will result in the need for more frequent instrument maintenance.

4.9 If hexadecane is added to waste samples or petroleum samples that are analyzed, some chromatographic peaks will elute after the target analytes. The oven temperature program

must include a post-analysis bake out period to ensure that semivolatile hydrocarbons are volatilized.

V. Safety

5.1 All samples submitted to an environmental laboratory should be treated as potential health hazards.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; therefore, each compound should be treated as a potential health hazard.

VI. Definitions

1. **BFB:** 4-Bromofluorobenzene.
2. **CCC:** Calibration check compound.
3. **D:** Drift.
4. **%D:** Percent drift.
5. **EICP:** Extracted ion current profile; a plot of ion abundance *vs.* time or scan number.
6. **EP:** Extraction procedure.
7. **FC-43:** Perfluoro-tri-N-butylamine.
8. **Field Sample:** All samples submitted by the client, including field quality control samples such as field blanks and trip blanks.
9. **GC:** Gas chromatograph.
10. **GC/MS:** Gas chromatograph/mass spectrometer system.
11. **Ion:** As used in this document, the *m/z* ratio.
12. **LCS:** Laboratory control sample; sometimes called a blank spike.
13. **MS:** Matrix spike.
14. **MSD:** Matrix spike duplicate.
15. **PTFE:** Polytetrafluoroethylene (Teflon®)
16. **QL:** Quantitation limit.
17. **Quality Control Sample:** Samples prepared at the laboratory for quality control purposes, including method blanks, matrix spikes, replicates, blank spikes, *etc*. Calibration standards are not included.
18. **RF:** Response factor.
19. **RPD:** Relative percent difference.
20. **RRT:** Relative retention time.

21. **RSD:** Relative standard deviation.
22. **%RSD:** percent relative standard deviation.
23. **SPCC:** System performance check compound.
24. **TCLP:** Toxicity characteristic leachate procedure.

VII. Sample Collection, Preservation and Handling

7.1 Using an appropriate sampling device, collect soil samples as soon as possible after the surface of the soil or other solid material has been exposed to the atmosphere.

7.2 Using the sample collection device, add about 5 grams of soil to a pre-weighed 40-mL, PTFE-lined screw cap, septum sealed vial containing 5 mL of methanol. It is required that the soil be completely covered by the methanol. Quickly brush off any soil on the vial threads and cap the vial securely. Store samples at 4 ^{o}C.

7.3 For dry weight determinations, a second 40-mL vial, which does not contain methanol, must be filled with sample.

Note: Each sampling group must be accompanied by a methanol trip blank for low level and high-level soil sampling.

VIII. Apparatus and Materials

8.1 Purge-and-trap device for aqueous samples - Described in Method 5030

8.2 Purge-and-trap device for solid samples - Described in Method 5035

8.3 Gas chromatography/mass spectrometer/data system

8.3.1 Gas chromatograph - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection with appropriate interface for sample introduction device. The system includes all required accessories, including syringes, analytical columns, and gases.

8.3.1.1 The GC should be equipped with variable constant differential flow controllers so that the column flow rate will remain constant throughout desorption and temperature program operation.

8.3.1.2 For some column configurations, the column oven must be cooled to less than 30°C, therefore, a sub-ambient oven controller may be necessary.

8.3.1.3 The capillary column is either directly coupled to the source or interfaced through a jet separator, depending on the size of the capillary and the requirements of the GC/MS system.

8.3.1.4 Capillary pre-column interface - This device is the interface between the sample introduction device and the capillary gas chromatograph and is

necessary when using cryogenic cooling. The interface condenses the desorbed sample components and focuses them into a narrow band on an uncoated fused-silica capillary pre-column. When the interface is flash heated, the sample is transferred to the analytical capillary column.

8.3.2 Gas chromatographic columns

GC Columns

Column	---	Restek, RTX-YMS, 40m x 0.18mm x 1um Restek, RTX-624, 75m x 0.53mm x 3um Restek, RTX-502.2 105m x 0.53mm x 3um
Carrier gas	---	helium

8.3.3 Mass spectrometer - Capable of scanning from 35 to 300 amu every 2 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for 4-Bromofluorobenzene (BFB), which meets all of the criteria in Table 4 when 5-50 ng of the GC/MS tuning standard (BFB) are injected through the GC. To ensure sufficient precision of mass spectral data, the desirable MS scan rate allows acquisition of at least five spectra while a sample component elutes from the GC.

8.3.4 An ion trap mass spectrometer may be used if it is capable of axial modulation to reduce ion-molecule reactions and can produce electron impact-like spectra that match those in the EPA/NIST Library. Because ion-molecule reactions with water and methanol in an ion trap mass spectrometer may produce interferences that coelute with chloromethane and chloroethane, the base peak for both of these analytes will be at m/z 49. This ion should be used as the quantitation ion in this case. The mass spectrometer must be capable of producing a mass spectrum for BFB, which meets all of the criteria in Table 3 when 5 or 50 ng are introduced.

8.3.5 GC/MS interface - Two alternatives may be used to interface the GC to the mass spectrometer.

8.3.5.1 Direct coupling, by inserting the column into the mass spectrometer, is generally used for 0.25-0.32 mm ID columns.

8.3.5.2 A jet separator, including an all-glass transfer line and glass enrichment device or split interface, is used with a 0.53 mm column.

8.3.5.3 Any enrichment device or transfer line may be used, if all of the performance specifications described in Sec. 8.0 (including acceptable calibration at 50 ng or less) can be achieved. GC/MS interfaces constructed entirely of glass or of glass-lined materials are recommended. Glass may be deactivated by silanizing with dichlorodimethylsilane.

8.3.6 Data system - A computer system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program must be interfaced to the mass spectrometer. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time and scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library should also be available.

8.4 Microsyringes - 10-, 25-, 100-, 250-, 500-, and 1,000-µL.

8.5 Syringe valve - Two-way, with Luer ends (three each), if applicable to the purging device.

8.6 Syringes - 5-10-, or 25-mL, gas-tight with shutoff valve.

8.7 Balance - Analytical, capable of weighing 0.0001 g, and top-loading, capable of weighing 0.1g.

8.8 Glass scintillation vials - 40-mL, with PTFE-lined screw-caps or glass culture tubes with PTFE-lined screw-caps

8.9 Disposable pipets - Pasteur

8.10 Volumetric flasks, Class A - 10-mL and 100-mL, with ground-glass stoppers

8.11 Spatula - Stainless steel

IX. Reagents

9.1 Reagent grade inorganic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all inorganic reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

9.2 Organic-free reagent water - All references to water in this method refer to organic-free reagent water, as defined in Chapter One, Method 8000.

9.3 Methanol, CH_3OH - Pesticide quality or equivalent, demonstrated to be free of analytes. Store away from other solvents.

9.4 Hydrochloric acid (1:1 v/v), HCI - Carefully add a measured volume of concentrated HCI to an equal volume of organic-free reagent water.

9.5 Stock solutions - Stock solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methanol, using assayed liquids or gases, as appropriate.

9.6 Stock solutions are typically prepared using purchased certified solutions, see table 4 and 5 for specific mixes prepared.

9.7 Transfer the stock standard solution into a bottle with a PTFE-lined screw-cap. Store, with minimal headspace and protected from light, at -10°C or less or as recommended by the standard manufacturer. Standards should be returned to the freezer as soon as the analyst has completed mixing or diluting the standards to prevent the evaporation of volatile target compounds.

9.7.1 Frequency of Standard Preparation

9.7.1.1 Standards for the permanent gases should be monitored frequently by comparison to the initial calibration curve. Fresh standards should be prepared if this check exceeds a 20% drift. Standards for gases usually need to be replaced after one week or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented. Dichlorodifluoromethane and dichloromethane will usually be the first compounds to evaporate from the standard and should, therefore, be monitored very closely when standards are held beyond one week.

9.7.1.2 Standards for the non-gases should be monitored frequently by comparison to the initial calibration. Fresh standards should be prepared if this check exceeds a 20% drift. Standards for non-gases usually need to be replaced after six months or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented. Standards of reactive compounds such as 2-chloroethyl vinyl ether and styrene may need to be prepared more frequently.

9.7.2 Optional Preparation of Calibration Standards from a Gas Mixture – An optional calibration procedure involves using a certified gaseous mixture daily, utilizing a commercially available gaseous analyte mixture of bromomethane, chloromethane, chloroethane, vinyl chloride, dichlorodifluoromethane and trichlorofluoromethane in nitrogen. Mixtures of documented quality are stable for as long as six months without refrigeration. (VOA-CYL III, RESTEK Corporation, Cat. #20194 or equivalent).

9.7.2.1 Before removing the cylinder shipping cap, be sure the valve is completely closed (turn clockwise). The contents are under pressure and should be used in a well-ventilated area.

9.7.2.2 Wrap the pipe thread end of the Luer fitting with PTFE tape. Remove the shipping cap from the cylinder and replace it with the Luer fitting.

9.7.2.3 Transfer half the working standard containing other analytes, internal standards, and surrogates to the purge apparatus.

9.7.2.4 Purge the Luer fitting and stem on the gas cylinder prior to sample removal using the following sequence:

a) Connect either the 100-µL or 500-µL Luer syringe to the inlet fitting of the cylinder.

b) Make sure the on/off valve on the syringe is in the open position.

c) Slowly open the valve on the cylinder and withdraw a full syringe volume.

d) Be sure to close the valve on the cylinder before you withdraw the syringe from the Luer fitting.

e) Expel the gas from the syringe into a well-ventilated area.

f) Repeat steps a through e one more time to fully purge the fitting.

9.7.2.5 Once the fitting and stem have been purged, quickly withdraw the volume of gas you require using steps 5.6.6.1.4(a) through (d). Be sure to close the valve on the cylinder and syringe before you withdraw the syringe from the Luer fitting.

9.7.2.6 Open the syringe on/off valve for 5 seconds to reduce the syringe pressure to atmospheric pressure. The pressure in the cylinder is 30 psi.

9.7.2.7 The gas mixture should be quickly transferred into the reagent water through the female Luer fitting located above the purging vessel.

NOTE: Make sure the arrow on the 4-way valve is pointing toward the female Luer fitting when transferring the sample from the syringe. Be sure to switch the 4-way valve back to the closed position before removing the syringe from the Luer fitting.

9.7.2.8 Transfer the remaining half of the working standard into the purging vessel. This procedure insures that the total volume of gas mix is flushed into the purging vessel, with none remaining in the valve or lines.

9.7.2.9 The concentration of each compound in the cylinder is typically 0.0025 µg/µL

9.7.2.10 The following are the recommended gas volumes spiked into 5 mL of water to produce a typical 5-point calibration:

Gas Volume	Calibration Concentration
40 µL	20 µg/L
100 µL	50 µg/L
200 µL	100 µg/L
300 µL	150 µg/
400 µL	200 µg/L

9.8 Secondary dilution standards - Using stock standard solutions, prepare secondary dilution standards in methanol containing the compounds of interest, either singly or mixed together. Secondary dilution standards must be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Store in a vial with no headspace. Replace after one week. Secondary

standards for gases should be replaced after one week unless the acceptability of the standard can be documented. When using premixed, certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations. The analyst should also handle and store standards as stated in Sec. 5.7 and return them to the freezer as soon as standard mixing or diluting is completed to prevent the evaporation of volatile target compounds.

9.9 Surrogate standards - The recommended surrogates are toluene-d_8, 4-bromofluorobenzene, 1,2-dichloroethane-d_4, and dibromofluoromethane. Other compounds may be used as surrogates, depending upon the analysis requirements. An internal standard/ surrogate spiking solution is purchased at a concentration of 250 µg/mL, in methanol. Each sample undergoing GC/MS analysis must be spiked with the spiking solution prior to analysis; this is performed by the autosampler in most cases. If a more sensitive mass spectrometer is employed to achieve lower detection levels, then more dilute surrogate solutions may be required.

9.10 Internal standards - The recommended internal standards are fluorobenzene, chlorobenzene-d_5, and 1,4-dichlorobenzene-d_4. Other compounds may be used as internal standards as long as they have retention times similar to the compounds being detected by GC/MS. An internal standard/surrogate spiking solution is purchased at a concentration of 250 µg/mL, in methanol. Each sample undergoing GC/MS analysis must be spiked with the spiking solution prior to analysis, this is performed by the autosampler in most cases. If a more sensitive mass spectrometer is employed to achieve lower detection levels, then more dilute internal standard solutions may be required. Area counts of the internal standard peaks should be between 50-200% of the areas of the target analytes in the mid-point calibration analysis.

9.11 4-Bromofluorobenzene (BFB) standard - A standard solution containing 25 ng/µL of BFB in methanol should be prepared. If a more sensitive mass spectrometer is employed to achieve lower detection levels, then a more dilute BFB standard solution may be required.

9.12 Calibration standards -There are two types of calibration standards used for this method: initial calibration standards and calibration verification standards. When using premixed certified solutions, store according to the manufacturer's documented holding time and storage temperature recommendations.

9.12.1 Initial calibration standards should be prepared at a minimum of five different concentrations from the secondary dilution of stock standards (see Secs. 5.7 and 5.8) or from a premixed certified solution. Prepare these solutions in organic-free reagent water. At least one of the calibration standards should correspond to sample concentration at or below that necessary to meet the data quality objectives of the project. The remaining standards should correspond to the range of concentrations found in typical samples but should not exceed the working range of the GC/MS system. Initial calibration standards should be mixed from fresh stock standards and dilution standards when generating an initial calibration curve.

9.12.2 Calibration verification standards should be prepared at a concentration near the mid-point of the initial calibration range from the secondary dilution of stock standards from

a premixed certified solution. Prepare these solutions in organic-free reagent water. See Sec. 7.4 for guidance on calibration verification.

9.12.3 All target analytes for a particular analysis should be included in the initial calibration and calibration verification standard(s). These target analytes may not include the entire list of analytes for which the method has been demonstrated. However, the laboratory shall not report a quantitative result for a target analyte that was not included in the calibration standard(s).

9.12.4 The calibration standards must also contain the internal standards chosen for the analysis.

9.13 Matrix spiking and laboratory control sample (LCS) standards - Matrix spiking standards should be prepared from volatile organic compounds which are representative of the compounds being investigated. At a minimum, the matrix spike should include 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. The matrix spiking solution should contain compounds that are expected to be found in the types of samples to be analyzed.

9.13.1 Some permits may require the spiking of specific compounds of interest, especially if polar compounds are a concern, since the spiking compounds listed above would not be representative of such compounds. The standard should be prepared in methanol, with each compound present at a concentration of 250 µg/10.0 mL.

9.13.2 The spiking solutions should not be prepared from the same standards as the calibration standards. However, the same spiking standard prepared for the matrix spike may be used for the LCS.

9.13.3 If a more sensitive mass spectrometer is employed to achieve lower detection levels, more dilute matrix spiking solutions may be required.

9.14 Great care must be taken to maintain the integrity of all standard solutions. It is recommended all standards in methanol be stored at -10°C or less, in amber bottles with PTFE-lined screw-caps.

X. Reagents

10.1 Instrument Performance Check Solution, 4-Bromofluorobenzene, 25.00 µg/mL in methanol

10.2 Matrix Spiking Standard Mix, 200 µg/mL each in methanol

10.3 Methanol, purge and trap grade

10.4 Purgeable Internal Standard/ Surrogate Standard Mix, 250 µg/mL each in methanol

10.5 8260B Liquid 54 Compound Mix, 2000 µg/mL each in methanol

10.6 8260B Gaseous Compounds Mix (6 components), 2000 µg/mL each in methanol

10.7 Various Independent Compounds, certified concentrations between 1000 to 20000 µg/mL each in methanol

10.8 Reagent Water: Deionized water in which no contamination is observed at or above the QL for any target compound

XI. Procedure

11.1 Various alternative methods are provided for sample introduction. All internal standards, surrogates, and matrix spiking compounds (when applicable) must be added to the samples before introduction into the GC/MS system. Consult the sample introduction method for the procedures by which to add such standards.

11.1.1 Direct injection - This includes: injection of an aqueous sample containing a very high concentration of analytes; injection of aqueous concentrates from Method 5031 (azeotropic distillation); and injection of a waste oil diluted 1:1 with hexadecane (Method 3585). Direct injection of aqueous samples (non-concentrated) has very limited applications. It is only used for the determination of volatiles at the toxicity characteristic (TC) regulatory limits or at concentrations in excess of 10,000 µg/L. It may also be used in conjunction with the test for ignitability in aqueous samples (along with Methods 1010 and 1020), to determine if alcohol is present at greater than 24%.

11.1.2 Purge-and-trap - This includes purge-and-trap for aqueous samples (Method 5030) and purge-and trap for solid samples (Method 5035). Method 5035 also provides techniques for extraction of high concentration solid and oily waste samples by methanol (and other water-miscible solvents) with subsequent purge-and-trap from an aqueous matrix using Method 5030.

11.1.2.1 The purge-and-trap of aqueous samples is performed at 40°C in addition to the soil/solid samples being performed at 40°C, to improve purging efficiency.

11.1.2.2 Aqueous and soil/solid samples may be purged at temperatures above those being recommended as long as all calibration standards, samples, and QC samples are purged at the same temperature, appropriate trapping material is used to handle the excess water, and the laboratory demonstrates acceptable method performance for the project. Purging of aqueous samples at elevated temperatures (e.g., 40°C) may improve the purging performance of many of the water soluble compounds, which have poor purging efficiencies at ambient temperatures.

11.1.3 Cartridge desorption - this technique may be for the introduction of volatile organics from sorbent cartridges (Method 5041) used in the sampling of air. The sorbent cartridges are from the volatile organics sampling train (VOST) or SMVOC (Method 0031).

11.2 Recommended chromatographic conditions

11.2.1 Instrument Setup

Purge Conditions	
Trap	Supelco, Vocarb 3000®
Purge gas	helium
Purge time	11 minutes
Purge flow rate	30 – 40 mL/minute
Purge temperature	40 °C

Desorb Conditions	
Temperature	260 °C
Flow rate	0 mL/minute
Time	1-2 minutes

Trap Initial Conditioning	
Temperature	265 °C
Flow rate	15-40 mL/minute (instrument dependent)
Time	90 minutes

Trap Reconditioning	
Temperature	265 °C
Flow rate	15 mL/minute
Time	8 minutes

Trap Reconditioning Between Analyses	
Temperature	265 °C
Flow rate	15-40 mL/minute (instrument dependent)

Time	8 minutes

11.2.2 Optimize purge and trap conditions for sensitivity and to minimize cross-contamination between samples. Once optimized, the same purge and trap conditions must be used for the analysis of all standards, field samples, and quality control samples.

11.2.3 Optimize GC conditions for analyte separation and sensitivity. Once optimized, the same GC conditions must be used for the analysis of all standards, quality control samples, and field samples.

11.3 Mass spectrometer

Electron energy	70 volts (nominal)
Mass range	35 – 300 amu
Scan time	0.6 - 2 sec/scan
Non-target reference library	EPA/ NIST

11.3.1 The GC/MS must be tuned to meet the manufacturer's specifications using FC-43.

11.3.1.1 Prior to the analysis of any standards, quality control samples, or field samples, the analyst must establish that the GC/MS meets the mass spectral ion abundance criteria for BFB. The BFB Performance Check Solution must be analyzed once at the beginning of each 12-hour period during which samples or standards are to be analyzed. The 12-hour tune period for GC/MS instrument performance check, calibration standards, and sample analysis begins at the moment of injection of the BFB analysis that the laboratory submits as documentation of a compliant instrument performance check. The time period ends after 12 hours has elapsed according to the system clock. The BFB performance check may be taken from the daily calibration standard.

11.3.1.2 Analyze 50 ng of BFB Performance Check Solution by direct injection or purge and trap using the gas chromatograph and mass spectrometer conditions specified above.

11.3.1.3 All subsequent standards, field samples, and quality control samples associated with a BFB analysis must use identical mass spectrometer instrument conditions.

11.3.1.4 The relative ion abundance criteria for BFB are listed in Table 2.

11.3.1.5 Internal and surrogate standards must be added to all standards, blanks, and samples. The recommended internal standards are fluorobenzene,

chlorobenzene-d_5, and 1,4-dichlorobenzene-d_4. The recommended surrogate standards are: toluene-d_8 4-bromofluorobenzene, 1,2-dichloroethane-d_4, and dibromofluoromethane

11.3.2 Each GC/MS system must be hardware-tuned to meet the criteria in Table 2 for a 50 ng injection or purging of 4-bromofluorobenzene (2-pL injection of the BFB standard). Analyses must not begin until these criteria are met.

11.3.2.1 In the absence of specific recommendations on how to acquire the mass spectrum of BFB from the instrument manufacturer, the following approach has been shown to be useful: The mass spectrum of BFB may be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of BFB. Do not perform background subtraction on any part of the BFB peak. Alternatively, the analyst may use other documented approaches suggested by the instrument manufacturer.

11.3.2.2 Use the BFB mass intensity criteria in Table 2 as tuning acceptance criteria. Alternatively, other documented tuning criteria may be used (e.g., CLP, Method 524.2, or manufacturer's instructions), provided that method performance is not adversely affected.

11.3.2.3 All subsequent standards, samples, MS/MSDs, LCSs, and blanks associated with a BFB analysis must use identical mass spectrometer instrument conditions.

11.3.3 Set up the sample introduction system as outlined in the method of choice (see Sec. *7.1)*. A different calibration curve is necessary for each method because of the differences in conditions and equipment. A set of at least five different calibration standards is necessary (see Sec. 5.12 and Method 8000). Calibration must be performed using the sample introduction technique that will be used for samples, or Method 5030, the purging efficiency for 5 mL of water is greater than for 25 mL. Therefore, develop the standard curve with whichever volume of sample that will be analyzed.

11.3.3.1 To prepare a calibration standard, add an appropriate volume of a secondary dilution standard solution to an aliquot of organic-free reagent water in a volumetric flask. Use a microsyringe and rapidly inject the alcoholic standard into the expanded area of the filled volumetric flask. Remove the needle as quickly as possible after injection. Mix by inverting the flask three times only. Discard the contents contained in the neck of the flask. Aqueous standards are not stable and should be prepared daily. Transfer 5.0 mL (or 25 mL if lower detection limits are required) of each

standard to a gas tight syringe along with 10 µL of internal standard. Then transfer the contents to the appropriate device or syringe. Some of the introduction methods may have specific guidance on the volume of calibration standard and the way the standards are transferred to the device.

11.3.3.2 The internal standards selected in Sec. 5.10 should permit most of the components of interest in a chromatogram to have retention--times of 0.80-1.20, relative to one of the internal standards. Use the base peak ion from the specific internal standard as the primary ion for quantitation (see Table 1). If interferences are noted, use the next most intense ion as the quantitation ion.

11.3.4 Proceed with the analysis of the calibration standards following the procedure in the introduction method of choice. For direct injection, inject 1 - 2 µL into the GC/MS system. The injection volume will depend upon the tolerance of the specific GC/MS system to water.

11.3.5 Tabulate the area response of the characteristic ions (see Table 5) against the concentration for each target analyte and each internal standard. Calculate response factors (RF) for each target analyte relative to one of the internal standards. The internal standard selected for the calculation of the RF for a target analyte should be the internal standard that has a retention time closest to the analyte being measured (Sec. 7.6.2).

The RF is calculated as follows:

$$\mathbf{RF} = \frac{\mathbf{A_s \times C_{is}}}{\mathbf{A_{is} \times C_s}}$$

where: A_s = Peak area (or height) of the analyte or surrogate

A_{is} = Peak area (or height) of the internal standard

C_s = Concentration of the analyte or surrogate

C_{is} = Concentration of the internal standard

11.3.6 System performance check compounds (SPCCs) - Calculate the mean RF for each target analyte using the five RF values calculated from the initial (5-point) calibration curve. A system performance check should be made before this calibration curve is used. Five compounds (the SPCCs) are checked for a minimum average response factor. These compounds are chloromethane; 1,1-dichloroethane; bromoform; chlorobenzene; and 1,1,2,2-tetrachloroethane. These compounds are used to check compound instability and to check for degradation caused by contaminated lines or active sites in the system. Example problems include:

11.3.6.1 Chloromethane is the most likely compound to be lost if the purge flow is

too fast.

11.3.6.2 Bromoform is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio relative to m/z 95 may improve bromoform response.

11.3.6.3 Tetrachloroethane and 1,1-dichloroethane are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

11.3.6.4 The minimum mean response factors for the volatile SPCCs are as follows:

Chloromethane	0.10
1, 1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1, 2, 2-Tetrachloroethane	0.30

11.3.7 Calibration check compounds (CCCs)

11.3.7.1 The purpose of the CCCs is to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may be indicative of system leaks or reactive sites on the column. Meeting the CCC criteria is not a substitute for successful calibration of the target analytes using one of the approaches described in Sec. 7.0 of Method 8000.

11.3.7.2 Calculate the standard deviation (SD) and relative standard deviation (RSD) of the response factors for all target analytes from the initial calibration, as follows:

$$SD = \sqrt{\frac{\sum_{n-1}^{n}\left(RF_i - \overline{RF}\right)^2}{n-1}}$$

$$\textbf{RSD} = \frac{\textbf{SD}}{\textbf{RF}} \textbf{ x 100}$$

where: $\overline{RF}$ = mean RF for each compound from the initial calibration
RF = RF for each of the calibration standards

n = Number of calibration standards, e.g., 5

11.3.7.3 The RSD should be less than or equal to 15% for each target analyte. However, the RSD for each individual Calibration Check Compound (CCC) must be equal or less than 30%. If the CCCs are not included in the list of analytes for a project, and therefore not included in the calibration standards, refer to Sec. 7.0 of Method 8000. The CCCs are:

1, 1-Dichloroethene	Toluene
Chloroform	Ethylbenzene
1 2-Dichloropropane	Vinyl chloride

11.3.7.4 If an RSD of greater than 30% is measured for any CCC, then corrective action to eliminate a system leak and/or column reactive sites is necessary before re-attempting calibration.

11.3.8 Evaluation of retention times - The relative retention times of each target analyte in each calibration standard should agree within 0.06 relative retention time units. Late-eluting compounds usually have much better agreement.

11.3.9 Linearity of target analytes

11.3.9.1 If the RSD of any target analyte is 15% or less, then the response factor is assumed to be constant over the calibration range, and the average response factor may be used for quantitation (Sec. 7.7.2).

11.3.9.2 If the RSD of any target analyte is greater than 15%, refer to Sec. 7.0 of Method 8000 for additional calibration options. One of the options must be applied to GC/MS calibration in this situation, or a new initial calibration must be performed.

NOTE: Method 8000 specifies a linearity criterion of 20% RSD. That criterion pertains to GC and HPLC methods other than GC/MS. Method 8260 requires 15% RSD as evidence of sufficient linearity to employ an average response factor.

11.3.9.3 When the RSD exceeds 15%, the plotting and visual inspection of a calibration curve can be a useful diagnostic tool. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.

11.4 GC/MS calibration verification - Calibration verification consists of three steps that are performed at the beginning of each 12-hour analytical shift.

11.4.1 Prior to the analysis of samples or calibration standards, inject or introduce 50 ng of the 4-bromofluorobenzene standard into the GC/MS system. The resultant mass

spectra for the BFB must meet the criteria given in Table 2 before sample analysis begins. These criteria must be demonstrated each 12-hour shift during which samples are analyzed.

11.4.2 The initial calibration curve (Sec. 7.3) for each compound of interest should be verified once every 12 hours prior to sample analysis, using the introduction technique used for samples. This is accomplished by analyzing a calibration standard at a concentration near the midpoint concentration for the calibrating range of the GC/MS. The results from the calibration standard analysis should meet the verification acceptance criteria provided in Secs. 7.4.4 through 7.4.7.

NOTE: The BFB and calibration verification standard may be combined into a single standard as long as both tuning and calibration verification acceptance criteria for the project can be met without interferences.

11.4.3 A method blank should be analyzed after the calibration standard, or at any other time during the analytical shift, to ensure that the total system (introduction device, transfer lines and GC/MS system) is free of contaminants. If the method blank indicates contamination then it is appropriate to analyze a solvent blank to demonstrate that the contamination is not a result of carryover from standards or samples. See Sec. 8.0 of Method 8000 for method blank performance criteria.

11.4.4 System Performance Check Compounds (SPCCs)

11.4.4.1 A system performance check must be made during every 12-hour analytical shift. Each SPCC compound in the calibration verification standard must meet its minimum response factor (see Sec. 7.3.5.4). This is the same check that is applied during the initial calibration.

11.4.4.2 If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before sample analysis begins.

11.4.5 Calibration Check Compounds (CCCs)

11.4.5.1 After the system performance check is met, the CCCs listed in Sec. 7.3.6 are used to check the validity of the initial calibration. Use percent difference when performing the average response factor model calibration. Use percent drift when calibrating using a regression fit model. Refer to Sec. 7.0 of Method 8000 for guidance on calculating percent difference and drift.

11.4.5.2 If the percent difference or drift for each CCC is less than or equal to 20%, the initial calibration is assumed to be valid. If the criterion is not met

(i.e., greater than 20% difference or drift), for any one CCC, then corrective action must be taken prior to the analysis of samples. If the CCC's are not included in the list of analytes for a project, and therefore not included in the calibration standards, then all analytes must meet the 20% difference or drift criterion. Calculate as follows:

$$\% \text{ Drift} = \frac{C_t - C_c}{C_t} * 100$$

$$\% \text{ Difference} = \left| \frac{RF_v - \text{mean RF}}{\text{mean RF}} \right| * 100$$

where: C_C = measured concentration
C_t = theoretical concentration
RF_v = verification standard response factor
Mean RF is from the initial calibration

11.4.5.3 Problems similar to those listed under SPCCs could affect the CCCs. If the problem cannot be corrected by other measures, a new five-point initial calibration must be generated. The CCC criteria must be met before sample analysis begins.

11.4.6 Internal standard retention time - The retention times of the internal standards in the calibration verification standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

11.4.7 Internal standard response - If the EICP area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to + 100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

11.5 GC/MS analysis of samples

11.5.1 All initial and re-analyses of samples must be performed within 14 days of sample collection if samples are preserved, 7 days if not preserved.

11.5.2 TCLP leachates are initially analyzed at a 1:5 dilution.

11.5.3 Water-Miscible Liquid Technique

11.5.3.1 All initial and re-analyses of samples must be performed within 14 days of sample collection.

11.5.3.2 Water-miscible liquids must be diluted at least 1:50 prior to analysis. Either of the following techniques may be used.

11.5.3.3 Transfer 2 mL of sample or sample dilution to a 100-mL volumetric flask and dilute to volume with reagent water. Transfer immediately to a 5-mL gas-tight syringe.

11.5.3.4 Prepare the dilution directly in the 5-mL gas-tight syringe by injecting at least 20 µL but no more than 100 µL into a syringe containing 5-mL of reagent water.

11.5.3.5 Continue the analysis as for water samples.

11.5.4 Low Concentration Soil/Sediment Technique

11.5.4.1 All initial and reanalysis of samples must be performed within 14 days of sample collection.

11.5.4.2 Analyze water-miscible wastes by the Water-Miscible Liquid Analysis technique.

11.5.4.3 To manually prepare the reagent water containing the surrogate spike compounds and the internal standards, remove the plunger from a 5-mL "Luerlock" type syringe equipped with a syringe valve and fill until overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 5.0 mL. Add 5 µL of 8260B Internal/Surrogate Spiking Solution to the syringe through the valve.

11.5.4.4 The sample for volatile organics consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Weigh 5 g of sample into a tared purge device. Use a top loading balance. Note and record the actual weight to the nearest 0.1 g.

11.5.4.5 If a 5-g sample will cause one or more target compounds to exceed the initial calibration range, the analyst may use a smaller aliquot but not less than 0.5 g. If a 0.5-g aliquot causes one or more target compounds to exceed the initial calibration range, the Medium/High Concentration Soil/Sediment Analysis technique must be used.

11.5.4.6 Immediately add the spiked reagent water to the purge device and connect the device to the purge and trap system.

11.5.4.7 After preparing the sample, weigh an additional 5-10 g aliquot of sample into a tared crucible. Determine the percent dry weight by drying

overnight at 105 ^{0}C. Allow it to cool in a desiccator before weighing. Concentrations of individual analytes will be reported relative to the dry weight of sediment.

$$\text{\% dry weight} = \frac{\text{g of dry sample}}{\text{g of wet sample}} * 100$$

11.5.4.8 Proceed with the analysis as outlined above for Water Sample Analysis starting with paragraph 7.

11.5.5 Medium/High Concentration Soil/Sediment Technique

11.5.5.1 All initial and reanalysis of samples must be performed within 14 days of sample collection.

11.5.5.2 Initial weight is recorded in the Methanol Prep Logbook for methanol preserved VOA vials prior to field sampling. Weigh the received preserved sample vial to determine the actual sample weight by subtracting the initial weight recorded from the total. Add any methanol to bring the ratio to 1:1 of sample to methanol. Apply a factor in cases of a low sample weight verses methanol.

11.5.5.3 The sample for volatile organics consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Weigh 5 g (wet weight) into a tared 20-mL vial. Use a top loading balance. Note and record the actual weight to the nearest 0.1 g.

11.5.5.4 Quickly add 5 mL of methanol. Cap and shake for 2 minutes. Let the suspended material settle.

11.5.5.5 These extracts must be stored in the dark at ≤ 6 °C prior to analysis.

11.5.5.6 Generally, a 1:50 dilution is performed on the extracts prior to analysis. If the extract is too concentrated to keep all target compounds within the initial calibration range, use an appropriate dilution in methanol.

11.5.5.7 After preparing the sample, weigh an additional 5-10 g aliquot of sample into a tared crucible. Determine the percent dry weight by drying overnight at 105 °C. Allow it to cool in a desiccator before weighing. Concentrations of individual analytes will be reported relative to the dry weight of sediment.

$$\text{\% dry weight} = \frac{\text{g of dry sample}}{\text{g of wet sample}} * 100$$

11.5.5.8 Proceed with the analysis as outlined above for Water Sample Analysis, starting with paragraph 7.

11.5.6 Method blank analysis

11.5.6.1 A volatile method blank must be prepared and analyzed each 12 hour shift, preferably after the calibration standards and after any high concentration samples.

11.5.6.2 The method blank is prepared in the same manner as samples, substituting appropriate blank material for the sample.

11.5.6.3 The method blank analysis must meet all the requirements of the Quality Control and Corrective Action section before field and other quality control samples can be analyzed. The concentration of each target compound must be less than it's required quantitation limit for the project.

11.5.7 LCS analysis

11.5.7.1 A volatile LCS should be prepared and analyzed at a frequency of 1 per batch of 20 samples per matrix, not to exceed 30 days between analyses.

11.5.7.2 The LCS is prepared by adding 12.5 µL of the 8260B MS/MSD Spiking Solution (see table 4) to 5 mL of reagent water. Internal standards/surrogate mixture is added by the autosampler or manually at the time of spiking.

11.5.7.3 Calculate the LCS recovery.

$$\textbf{\% Recovery} = \frac{C_S}{C_A} * 100$$

where: C_S = analyte concentration recovered
C_A = concentration of analyte added

11.5.7.4 The LCS should meet the recovery range requirements determined by the laboratory. Although no action is required, frequent failure to meet these limits indicates a problem in the analytical system that should be investigated.

11.5.8 MS/MSD analysis

11.5.8.1 A volatile MS/MSD must be prepared and analyzed at a frequency of 1 per 20 samples per matrix prepared by the same technique, not to exceed 30 days between analysis.

11.5.8.2 If the client has designated a sample to be used for the MS/MSD, this sample must be spiked. Otherwise, a field sample (not a quality control or

performance evaluation sample) shall be chosen by the analyst. A known blank (field rinse blank or like) must not be used as a spike sample.

11.5.8.3 Prepare the MS/MSD by adding 12.5 ul of the 8260B MS/MSD Spiking Solution 8260B (see table 4) at a concentration of 200ppm to each of two aliquots of the sample chosen for spiking. Internal standards/surrogate mixture is added by the autosampler or manually at the time of spiking. The sample volume used for the MS/MSD must be the same as used for the unspiked sample analysis.

11.5.8.4 Calculate the MS and MSD recoveries.

$$\%Rec = \frac{C_S - C_U}{C_A} * 100$$

where: C_S = analyte concentration in spiked sample
C_U = analyte concentration in unspiked sample
C_A = Concentration of analyte added

11.5.8.5 Calculate the percent recovery and RPD for the MS/MSD.

$$\%Recovery = \frac{\text{spiked sample result - sample result}}{\text{spike added}} * 100$$

$$RPD = \frac{\text{matrix spike recovery - matrix spike duplicate recovery}}{1/2 * (\text{matrix spike recovery + matrix spike duplicate recovery})} * 100$$

11.5.8.6 The MS/MSD must meet the requirements in the Quality Control and Corrective Action section. The quality control limits for MS/MSD recovery are generated by the laboratory on an annual basis and are advisory. Although no action is required, frequent failure to meet these limits indicates a problem in the analytical system that should be investigated. If an MS/MSD fails to meet the established criteria, an LCS must be analyzed to determine if the sample is exhibiting matrix interference or if the system is out of control.

11.5.9 BFB tuning criteria and GC/MS calibration verification criteria must be met before analyzing samples.

11.5.10 All samples and standard solutions must be allowed to warm to ambient temperature before analysis. Set up the introduction device as outlined in the method of choice.

11.5.11 The process of taking an aliquot destroys the validity of remaining volume of an aqueous sample for future analysis. Therefore, if only one VOA vial is provided to

the laboratory, the analyst should prepare two aliquots for analysis at this time, to protect against possible loss of sample integrity. This second sample is maintained only until such time when the analyst has determined that the first sample has been analyzed properly. For aqueous samples, one 20-mL syringe could be used to hold two 5-mL aliquots. If the second aliquot is to be taken from the syringe, it must be analyzed within 24 hours. Care must be taken to prevent air from leaking into the syringe.

11.5.12 Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample or standard bottle, which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. If lower detection limits are required, use a 25-mL syringe, and adjust the final volume to 25.0 mL.

11.5.13 The following procedure may be used to dilute aqueous samples for analysis of volatiles. All steps must be performed without delays, until the diluted sample is in a gas-tight syringe or alternately be performed by the autosampler.

11.5.13.1 Dilutions may be made in volumetric flasks (10- to 100-mL). Select the volumetric flask that will allow for the necessary dilution. Intermediate dilution steps may be necessary for extremely large dilutions.

11.5.13.2 Calculate the approximate volume of organic-free reagent water to be added to the volumetric flask, and add slightly less than this quantity of organic-free reagent water to the flask.

11.5.13.3 Inject the appropriate volume of the original sample from the syringe into the flask. Aliquots of less than 1 mL are not recommended. Dilute the sample to the mark with organic-free reagent water. Cap the flask, invert, and shake three times. Repeat above procedure for additional dilutions.

11.5.13.4 Fill a 5-mL syringe with the diluted sample, as described in Sec. 7.5.5.

11.5.14 Compositing aqueous samples prior to GC/MS analysis

11.5.14.1 Add 5 mL of each sample (up to 5 samples are allowed) to a 25-mL glass syringe. Special precautions must be made to maintain zero headspace in the syringe. Larger volumes of a smaller number of samples may be used, provided that equal volumes of each sample are composited.

11.5.14.2 The samples must be cooled to 4°C or less during this step to minimize volatilization losses. Sample vials may be placed in a tray of ice during the processing.

11.5.14.3 Mix each vial well and draw out a 5-mL aliquot with the 25-mL syringe.

11.5.14.4 Once all the aliquots have been combined on the syringe, invert the

syringe several times to mix the aliquots. Introduce the composited sample into the instrument, using the method of choice (see Sec. 7.1).

11.5.14.5 If less than five samples are used for compositing, a proportionately smaller syringe may be used, unless a 25-mL sample is to be purged.

11.5.15 Add 5 µL of the combined surrogate/internal standard spiking solution to each sample either manually or by autosampler. The surrogate and internal standards are mixed and added as a single spiking solution. The addition of 5 µL of the surrogate spiking solution to 5 mL of aqueous sample will yield a concentration of 50 µg/L of each surrogate standard. The addition of 5 µL of the surrogate spiking solution to 5 g of a non-aqueous sample will yield a concentration of 50 µg/kg of each standard. If a more sensitive mass spectrometer is employed to achieve lower detection levels more dilute surrogate and internal standard solutions may be required. The sample and spike amounts may vary dependent upon the equipment requirements however, the spike levels remain the same as prescribed.

11.5.16 Add 5 µL of the matrix spike solution to a 5-mL aliquot of the sample chosen for spiking. Disregarding any dilutions, this is equivalent to a concentration of 50 µg/L of each matrix spike standard.

11.5.16.1 Follow the same procedure in preparing the laboratory control sample (LCS), except the spike is added to a clean matrix. See Sec. 8.4 and Method 5000 for more guidance on the selection and preparation of the matrix spike and the LCS.

11.5.16.2 If a more sensitive mass spectrometer is employed to achieve lower detection levels, more dilute matrix spiking and LCS solutions may be required.

11.5.17 Analyze the sample following the procedure in the introduction method of choice.

11.5.17.1 For direct injection, inject 1 to 2 µL into the GC/MS system. The volume limitation will depend upon the chromatographic column chosen and the tolerance of the specific GC/MS system to water (if an aqueous sample is being analyzed).

11.5.17.2 The concentration of the internal standards, surrogates, and matrix spiking standards (if any) added to the injection aliquot must be adjusted to provide the same concentration in the 1-2 µL injection as would be introduced into the GC/MS by purging a 5-mL aliquot.

NOTE: It may be a useful diagnostic tool to monitor internal standard retention times and responses (area counts) in all samples, spikes, blanks, and standards to effectively check drifting method performance, poor injection execution, and anticipate the need for system inspection and/or maintenance.

11.5.18 If the initial analysis of the sample or a dilution of the sample has a concentration of any analyte that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. Secondary ion quantitation is allowed only when there are sample interferences with the primary ion.

11.5.18.1 When ions from a compound in the sample saturate the detector, this analysis must be followed by the analysis of an organic-free reagent water blank. If the blank analysis is not free of interferences, then the system must be decontaminated. Sample analysis may not resume until the blank analysis is demonstrated to be free of interferences.

11.5.18.2 All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

11.5.19 The use of selected ion monitoring (SIM) is acceptable in situations requiring detection limits below the normal range of full El spectra. However, SIM may provide a lesser degree of confidence in the compound identification unless multiple ions are monitored for each compound.

11.5.20 The qualitative identification of each compound determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met.

11.5.20.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

11.5.20.2 The relative retention time (RRT) of the sample component is within ± 0.06 RRT units of the RRT of the internal standard component.

11.5.20.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)

11.5.20.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC

retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

11.5.20.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

11.5.20.6 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes coelute (i.e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

11.5.21 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

11.5.21.1 For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Use the following guidelines for making tentative identifications:

1. Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.
2. The relative intensities of the major ions should agree within ± 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).
3. Molecular ions present in the reference spectrum should be present in the sample spectrum.
4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

11.6 Quantitative analysis

11.6.1 Once a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. The internal standard used shall be the one nearest the retention time of that of a given analyte.

11.6.2 If the RSD of a compound's response factors is 15% or less, then the concentration in the extract may be determined using the average response factor (RF) from initial calibration data (7.3.6). See Method 8000, Sec. 7.0, for the equations describing internal standard calibration and either linear or non-linear calibrations.

11.6.3 Where applicable, the concentration of any non-target analytes identified in the sample (Sec. 7.6.2) should be estimated. The same formulae should be used with the following modifications: The areas A_x and A_{is} should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1.

11.6.4 The resulting concentration should be reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

XII. Quality Control

12.1 Refer to Chapter One and Method 8000 for specific quality control (QC) procedures. Quality control procedures to ensure the proper operation of the various sample preparation and/or sample introduction techniques can be found in Methods 3500 and 5000. The maintain records to document the quality of the data generated according to laboratory's Quality Manual.

12.2 Quality control procedures necessary to evaluate the GC system operation are found in Method 8000, Sec. 7.0 and include evaluation of retention time windows, calibration verification and chromatographic analysis of samples. In addition, instrument QC requirements may be found in the following sections of Method 8260:

12.2.1 The GC/MS system must be tuned to meet the BFB specifications in Secs. 7.3.1 and 7.4.1.

12.2.2 There must be an initial calibration of the GC/MS system as described in Sec. 7.3.

12.2.3 The GC/MS system must meet the SPCC criteria described in Sec. 7.4.4 and the CCC criteria in Sec. 7.4.5, each 12 hours.

12.3 Initial Demonstration of Proficiency - Each laboratory must demonstrate initial proficiency

with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat the following operations whenever new staff is trained or significant changes in instrumentation are made. See Method 8000, Sec. 8.0 for information on how to accomplish this demonstration.

12.4 Sample Quality Control for Preparation and Analysis – Documentation is required demonstrating the effect of the matrix on method performance (precision, accuracy, and detection limit). At a minimum, this includes the analysis of QC samples including a method blank, matrix spike, a duplicate, and a laboratory control sample (LCS) in each analytical batch and the addition of surrogates to each field sample and QC sample.

12.4.1 Before processing any samples, the analyst should demonstrate through the analysis of a method blank that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is analyzed or there is a change in reagents, a method blank should be analyzed as a safeguard against chronic laboratory contamination. The blanks should be carried through all stages of sample preparation and measurement.

12.4.2 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on knowledge of the samples in the sample batch. If samples are expected to contain target analytes, then laboratories may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, laboratories should use a matrix spike and matrix spike duplicate pair.

12.4.3 A Laboratory Control Sample (LCS) must be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

12.5 Surrogate recoveries - The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. See Method 8000, Sec. 8.0 for information on evaluating surrogate data and developing and updating surrogate limits.

12.6 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the calibration verification standard is to be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably, the injector is leaking, the injector

septum needs replacing, etc. If any changes are made to the system (e.g., the column changed), recalibration of the system must take place.

XIII. Method Performance

13.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects. Water and Solid MDLs were calculated using the appropriate preparation methods and are presented in Table 1.

13.2 The following chart outlines the quality control and corrective action requirements for this procedure. Where re-extraction or re-analysis is indicated, this must be performed within the applicable holding time.

Quality Control Problem	Corrective Action
BFB ion abundance criteria outside acceptance windows (see Table 2)	Retune and recalibrate the GC/MS. It may be necessary to clean the ion source before retuning.

The response factors of the SPCCs do not meet the acceptance criteria	Recalibrate the GC/MS. It may be necessary to clean the source, change the column, or take other corrective action before recalibrating.
	• Low response for chloromethane usually indicates that the purge rate is too fast
	• Low response for bromoform may be caused by
	  ◦ Purge rate too slow
	  ◦ Cold spots or active sites in the transfer lines
	  ◦ Relative abundance of the high m/z ions (174 and 176) too low
	• Low response for 1,1,2,2-tetrachloroethane and/or 1,1-dichloroethane usually indicates contaminated transfer lines or active sites in the trap materials

Initial calibration CCCs %RSD outside acceptance window	Check for system leaks or active sites and re-calibrate the instrument.
Continuing calibration RF and/or %D outside acceptance window	See the corrective action above for the initial calibration
Continuing calibration internal standard retention time not within ±30 seconds of the mid-point level internal standards of the most recent initial calibration	Check the GC system for malfunction. Perform a new initial or continuing calibration and reanalyze all samples analyzed with the failed standard.
Continuing calibration internal standard areas not within -50% to + 100% of the	• Check for calculation error. If an error is found, correct the calculations and verify that the recalculated responses meet

Quality Control Problem	Corrective Action
mid-point level internal standards of the most recent initial calibration	the acceptance criteria. • Check for mass spectrometer malfunction. If a problem is found, correct the malfunction, then retune, recalibrate, and reanalyze the affected samples.
Method blank contamination ≥ QL	The method blank and all associated samples must be reanalyzed.
Method blank surrogate recovery outside acceptance window	Reanalyze the method blank and all associated samples.
Interference with primary quantitation ion	Use a secondary quantitation ion listed in Table 1 and document problem in Case Narrative.
Quantitation ion saturates detector	Standards: Adjust the analytical system to eliminate saturation while maintaining sufficient sensitivity to meet QL requirements. Retune and perform a new initial calibration.
	Field and quality control samples: Dilute and reanalyze.
Sample internal standard retention times not within ± 30 seconds the mid-point level internal standards of the most recent initial calibration	Check the system for malfunction and re-analyze the affected samples.
Sample internal standard areas not within a factor of (-50% to +100%) of associated continuing calibration standard	**All analyses:** Check for calculation error. If an error is found, correct the calculations and verify that the recalculated responses meet the acceptance criteria.
	All analyses: Check for mass spectrometer malfunction. If a problem is found, correct the malfunction, retune, recalibrate, and reanalyze the affected samples.
	Field samples and MS/MSD: Reanalyze the sample. If the internal standard area criteria are met, the original analysis is invalid and only the reanalysis is to be submitted. If the criteria are not met, matrix interference is assumed and both analyses are to be submitted. **NOTE:** For the sample selected for MS/MSD, the MS and MSD serve as reanalysis for this purpose. All three analyses must confirm or reject the matrix interference assumption; otherwise, reanalysis is required to resolve the situation. Submit only the valid analyses. Document matrix interference in the Case Narrative.
RRT for surrogate not within ± 0.06 RRT units of RRT in the associated calibration standard	Check the GC system for malfunction. Reanalyze all failed standards, field samples, and quality control samples.
Surrogate recovery outside acceptance window	• Check for calculation error. If an error is found, correct the calculations and verify that the recalculated recoveries meet

Quality Control Problem	Corrective Action
	the acceptance criteria. • If no calculation error is found, reanalyze the sample. If the surrogate recovery criteria are met, the original analysis is invalid and only the reanalysis is to be submitted. If the criteria are not met, matrix interference is assumed. NOTE: For the sample selected for MS/MSD, the MS and MSD serve as re-extractions and re-analyses for this purpose. All three analyses must confirm or reject the matrix interference assumption; otherwise, re-extraction and reanalysis is required to resolve the situation. Submit only the valid analyses. Document matrix interference in the Case Narrative.

Table 1:

CAS No.	Compound	Primary Ion	Secondary Ion	Water PQL ug/L	Soil PQL ug/kg
67-64-1	Acetone	43	58	10	20
107-02-8	Acrolein	56	55	20	20
107-13-1	Acrylonitrile	53	52	20	20
71-43-2	Benzene	78	77	5	5
108-86-1	Bromobenzene	156	77	5	5
74-97-5	Bromochloromethane	128	130	5	5
75-27-4	Bromodichloromethane	83	85	5	5
75-25-2	Bromoform	173	171	5	5
74-83-9	Bromomethane	94	96	10	10
78-93-3	2-Butanone (MEK)	43	72	10	10
104-51-8	n-Butylbenzene	134	91	5	5
135-98-8	sec-Butylbenzene	105	134	5	5
98-06-6	tert-Butylbenzene	134	91	5	5
75-15-0	Carbon disulfide	76		5	5
56-23-5	Carbon tetrachloride	117	119	5	5
108-90-7	Chlorobenzene	112	77	5	5
75-00-3	Chloroethane	64	66	10	10
110-75-8	2-Chloroethyl vinyl ether	63	106	5	5
67-66-3	Chloroform	83	85	5	5
74-87-3	Chloromethane	50	52	10	10
95-49-8	2-Chlorotoluene	91	126	5	5
106-43-4	4-Chlorotoluene	91	126	5	5
96-12-8	1,2-Dibromo-3-chloropropane	75	155	5	5
124-48-1	Dibromochloromethane	129	127	5	5
106-93-4	1,2-Dibromoethane (EDB)	109	107	5	5
74-95-3	Dibromomethane	93	95	5	5
95-50-1	1,2-Dichlorobenzene	146	111	5	5
541-73-1	1,3-Dichlorobenzene	146	111	5	5
106-46-7	1,4-Dichlorobenzene	146	111	5	5
75-71-8	Dichlorodifluoromethane	85	87	10	10
75-34-3	1,1-Dichloroethane	63	65	5	5
107-06-2	1,2-Dichloroethane	62	64	5	5
75-35-4	1,1-Dichloroethene	96	61	5	5
156-59-4	cis-1,2-Dichloroethene	96	61	5	5
156-60-5	trans-1,2-Dichloroethene	96	61	5	5
78-87-5	1,2-Dichloropropane	63	62	5	5
142-28-9	1,3-Dichloropropane	76	78	5	5
590-20-7	2,2-Dichloropropane	77	97	5	5
563-58-6	1,1-Dichloropropene	75	110	5	5
10061-01-5	cis-1,3-Dichloropropene	75	77	5	5
10061-02-6	trans-1,3-Dichloropropene	75	77	5	5
100-41-4	Ethylbenzene	91	106	5	5
87-68-3	Hexachlorobutadiene	225	227	5	5

CAS No.	Compound	Primary Ion	Secondary Ion	Water PQL ug/L	Soil PQL ug/kg	
591-78-6	2-Hexanone	58	43	10	10	
98-82-8	Isopropylbenzene	105	120	5	5	
99-87-6	4-Isopropyltoluene	119	134	5	5	
1634-04-4	Methyl tert-butyl ether (MTBE)	73	57	5	5	
108-10-1	4-Methyl-2-pentanone (MIBK)	43	58	10	10	
75-09-2	Methylene chloride	84	86	5	5	
91-20-3	Naphthalene	128	129	5	5	
103-65-1	n-Propylbenzene	91	120	5	5	
100-42-5	Styrene	104	78	5	5	
96-18-4	1,2,3-Trichloropropane	110	77	5	5	
630-20-6	1,1,1,2-Tetrachloroethane	133	131	5	5	
79-34-5	1,1,2,2-Tetrachloroethane	83	131	5	5	
127-18-4	Tetrachloroethene (PCE)	164	166	5	5	
108-88-3	Toluene	92	91	5	5	
87-61-6	1,2,3-Trichlorobenzene	180	182	5	5	
120-82-1	1,2,4-Trichlorobenzene	180	182	5	5	
71-55-6	1,1,1-Trichloroethane	97	99	5	5	
79-00-5	1,1,2-Trichloroethane	97	99	5	5	
79-01-6	Trichloroethene (TCE)	130	132	5	5	
75-69-4	Trichlorofluoromethane	101	103	10	10	
76-13-1	Trichlorotrifluoroethane	151	153	10	10	
95-63-6	1,2,4-Trimethylbenzene	105	120	5	5	
108-67-8	1,3,5-Trimethylbenzene	105	120	5	5	
108-05-4	Vinyl acetate	43	86	10	10	
75-01-4	Vinyl chloride	62	64	10	10	
95-47-6	o-Xylene	91	106	5	5	
133-02-07	m,p-Xylenes	91	106	5	5	
107060-07-0	1,2-Dichloroethane-d_4 (S)	67	65	5	5	
462-06-6	Fluorobenzene (IS)	96	77	5	5	
3114-55-4	Chlorobenzene-d_5 (IS)	117		5	5	
3855-82-1	1,4-Dichlorobenzene-d_4 (IS)	152		5	5	
353-55-9	Dibromofluoromethane (S)	113		5	5	
2037-26-5	Toluene-d_8 (S)	98		5	5	
460-00-4	4-Bromofluorobenzene (S)	176	174	5	5	

Table 2: Ion Abundance Criteria for BFB

Mass (m/z)	Required Relative Abundance
50	15% to 40% of mass 95
75	30% to 60% of mass 95
95	Base peak, 100% relative abundance
96	5% to 9% of mass 95
173	Less than 2% of mass 174
174	Greater than 50% of mass 95
175	5% to 9% of mass 174
176	Greater than 95% of mass 174 but less than 101% of mass 174
177	5% to 9% of mass 176

Table 3: Acceptance Criteria for Initial and Continuing Calibration

Compound		Initial Calibration		Continuing Calibration	
		Min. Avg. RF	Max. % RSD	Min. RF	Max. % D
Bromoform	SPCC	0.100	---	0.100	---
Chlorobenzene	SPCC	0.300	---	0.300	---
Chloroform	CCC	---	30	---	20
Chloromethane	SPCC	0.100	---	0.100	---
1,1-Dichloroethane	SPCC	0.100	---	0.100	---
1,1-Dichloroethene	CCC	---	30	---	20
1,2-Dichloropropane	CCC	---	30	---	20
Ethylbenzene	CCC	---	30	---	20
1,1,2,2-Tetrachloroethane	SPCC	0.300	---	0.300	---
Toluene	CCC	---	30	---	20
Vinyl chloride	CCC	---	30	---	20

Table 4: 8260 Second Source Spike Standards

Compound	Initial Stock Concentration	Initial Amount Stock Used	Final Volume	Final Concentration
8260 SPIKE MIX 1				
MTBE	2000ppm	100ul	1000ul	200ppm
Ketones	2000ppm	100ul	1000ul	200ppm
Vinyl Acetate	2000ppm	100ul	1000ul	200ppm
Carbon Disulfide	2000ppm	100ul	1000ul	200ppm
2-CEVE	2000ppm	100ul	1000ul	200ppm
54 Comp.	2000ppm	100ul	1000ul	200ppm
Acrylonitrile	20,000ppm	10ul	1000ul	200ppm
Methacrylonitrile	2000ppm	100ul	1000ul	200ppm
8260 SPIKE MIX 2				
1,1,2 TCTFE	2000ppm	100ul	1000ul	200ppm
Diethyl ether	5000ppm	40ul	1000ul	200ppm
6 Comp. Gas Mix	2000ppm	100ul	1000ul	200ppm
Tetrahydrofuran	2000ppm	100ul	1000ul	200ppm
Ketones	2000ppm	100ul	1000ul	200ppm

Table 5: 8260B Calibration Standards Mix

Compound	Initial Stock Concentration	Initial Amount Stock Used	Final Volume	Final Concentration
MIX 1				
MTBE	2000ppm	100ul	1000ul	200ppm
Ketones	2000ppm	100ul	1000ul	200ppm
Acrolein	20,000ppm	10ul	1000ul	200ppm
Acrylonitrile	20,000ppm	10ul	1000ul	200ppm
Carbon Disulfide	20000ppm	10ul	1000ul	200ppm
Vinyl Acetate	20,000ppm	10ul	1000ul	200ppm
Diethyl ether	20,000ppm	10ul	1000ul	200ppm
MIX 2				
2-CEVE	1000ppm	200ul	1000ul	200ppm
1,1,2 TCTFE	1000ppm	100ul	1000ul	200ppm
Tetrahydrofuran	20,000ppm	10ul	1000ul	200ppm
Methacrylonitrile	20,000ppm	10ul	1000ul	200ppm
54 Component	2000ppm	100ul	1000ul	200ppm
6 Comp. Gas Mix	2000ppm	100ul	1000ul	200ppm

SOP Revision History

Date	Previous Revision No.	New Revision No.	Description of Changes	Effective Date	Initiated by
8/5/09	2.21	2.3	Added revision history table	8/5/09	LM
9/23/11	2.3	2.4	Changed format	9/23/11	LM

Total Kjeldahl Nitrogen

EPA 351.2

Prepared by: Melisa Montgomery, Quality Assurance Officer

Approved by: Robert Stevenson, Laboratory Director

Reviewed and Implemented by: Ronald Warila, General Manager

Reference

Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1983, method 351.2 and 351.1

Determination of Total Kjeldahl Nitrogen by Flow Injection Analysis, Method 10-107-06-2-D, Lachat Instruments, Inc

Technical Report EPA/ CE-81-1, Procedures for Handling and Chemical Analysis of Sediment and Water Samples, May 1981

I. Applicability

1.1 Analyte: Total Kjeldahl Nitrogen

1.2 Matrix: Water, wastewater, soil, sludge, and waste extracts

1.3 Regulation: NPDES, CWA

1.4 The applicable range is 0.5 to 20.00 mg N/L.

1.5 The method detection limit is 0.5 mg N/L.

1.6 The method throughput is 60-80 injections per hour.

II. Method Summary

2.1 The sample is heated in the presence of sulfuric acid (H_2SO_4) for two and one half hours. The residue is cooled, diluted with water and analyzed for ammonia. This digested sample may also be used for phosphorus determination.

2.2 Total Kjeldahl nitrogen is the sum of free-ammonia and organic nitrogen compounds which are converted to ammonium sulfate $(NH_4)_2SO_4$, under the conditions of the digestion described.

2.3 Organic Nitrogen may be reported by subtracting the ammonia results (determined by method 350.1) in mg/L from the TKN results in mg/L for a sample.

2.4 Total Nitrogen may be reported by adding the TKN results in mg/L to the combined Nitrate and Nitrite results in mg/L (determined by method SM4500-NO_3F).

2.5 Approximately 0.3 ml. of the digested sample is injected onto the chemistry manifold where its pH is controlled by raising it to a known, basic pH by neutralization with a concentrated buffer. This in-line neutralization converts the ammonium cation to ammonia, and also prevents undue influence of the sulfuric acid matrix on the pH-sensitive color reaction that follows.

2.6 The ammonia thus produced is heated with salicylate and hypochlorite to produce blue color which is proportional to the ammonia concentration. The color is intensified by adding sodium nitroprusside. The presence of potassium tartrate in the buffer prevents precipitation of calcium and magnesium.

III. Interferences

3.1 Samples must not consume more than 10% of the sulfuric acid during the digestion. The buffer (reagent 3) will only accommodate 4.5-5.0% sulfuric acid without any significant change in signal intensity.

3.2 High nitrate concentrations >10 times the TKN level will suppress the TKN results. A dilution must be performed prior to digestion to eliminate the effect.

3.3 All final digestates must be free of turbidity, filter if necessary.

IV. Definitions

4.1 **Calibration Blank (CB)** -- A volume of reagent water in the same matrix as the calibration standards, but without the analyte.

4.2 **Calibration Standard (CAL)** -- A solution prepared from the primary dilution standard solution or stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

4.3 **Instrument Performance Check Solution (IPC)** - A solution of one or more method analytes used to evaluate the performance of the instrument system with respect to a defined set of criteria.

4.4 **Laboratory Fortified Blank (LFB)** -- an aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

4.5 **Laboratory Fortified Matrix (LFM)** -- An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.

4.6 **Laboratory Reagent Blank (LRB)**—An aliquot of reagent water or other blank matrices that is digested exactly as a sample in including exposure to all glassware, equipment, and reagents that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents or the apparatus.

4.7 **Linear Calibration Range (LCR)** -- The concentration range over which the instrument response is linear.

4.8 **Method Detection Limit (MDL)** — The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

4.9 **Quality Control Sample (QCS)** -- A solution of method analytes of known concentrations that is used to spike an aliquot of LRB or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards.

4.10 **Stock Standard Solution (SSS)** -- A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

V. Procedure for Distillation

5.1 **Important:** If the block digester tubes are not completely dry and have water droplets on them, there exists the possibility of ammonia contamination in the water droplets. Ensure the tubes are completely dry before beginning the digestion procedure.

5.2 To 20.0 mL of sample or QC standard, add 5 mL digestion solution and mix thoroughly.

5.3 The following QC standards must be digested with each batch of 20 samples or less:

20 mg/L standard
5 mg/L standard
0.5 mg/L standard
10 mg/L LCS
Method blank

5.4 Add 2 - 4 Alundum granules or 5-6 Teflon stones to each tube for smooth boiling

5.5 Verify that boiling stones have been placed in each tube. Place tubes in the preheated digestion block for one hour at 160 °C. Water from the sample should have boiled off before increasing the temperature.

5.6 Ramp the digestion block up to 380 °C and set the timer at 90 minutes. The typical ramp time is 50 - 60 minutes. **The temperature must be maintained at 380 °C for 30 minutes.**

5.7 Before removing samples, gather the necessary supplies to dilute the samples with water.

5.7.1 Remove the samples from the block and **allow only *5* minutes cooling**.

5.7.2 Add water to the samples rapidly so that all samples are diluted within 10 minutes of removal from the block.

5.8 Add 20.0 mL DI water to each tube and vortex to mix. The longer the samples have been allowed to cool, the longer the samples should be vortexed.

5.9 Transfer sample to a polypropylene snap-cap vial. Filter out any turbidity, if applicable, only after being vortexed.

VI. Colorimetric Analysis Procedure

6.1 Setup the manifold as shown in diagram 1.

6.2 Pump DI water through all reagent lines and check for leaks and smooth flow. Switch to reagents and allow the system to equilibrate until a stable baseline is achieved.

6.3 Verify input peak timing and integration window parameters using the green dye provide by the manufacturer if necessary followed by DI water flush.

6.4 Place standards in the autosampler, and fill the sample tray. Input the information required by data system, such as concentration, replicates and QC scheme.

6.5 **Add buffer line first, pump for *5* minutes before adding the rest of the reagents.**

6.6 Calibrate the instrument by injecting the standards. The data system will then associate the

concentrations with responses for each standard.

6.7 After the calibration has been established, it must be verified by the analysis of a suitable quality control sample (QCS).

6.7.1 If measurements exceed +/-10% of the established QCS value, the analysis should be terminated and the instrument re-calibrated.

6.7.2 The new calibration must be verified before continuing analysis. Periodic reanalysis (every 10 samples or less) of the QCS can be substituted for continuing calibration check.

6.8 After a stable baseline has been obtained, start the sampler and perform analysis.

6.9 Important Notes

6.9.1 Allow at least 15 minutes for the heating unit to warm up to 60 °C.

6.9.2 If sample concentrations are greater than the high standard the digested sample should be diluted with Reagent 7. When the auto diluter is used, Reagent 7 should be used as diluent. **Do not dilute digested samples or standards with DI water.**

6.9.3 If the salicylate reagent is merged with a sample containing sulfuric acid in the absence of the buffer solution, the salicylate reagent will precipitate. If this occurs all Teflon manifold tubing should be replaced, alternately if flow is only partially restricted, flush the system with 50% sodium hydroxide to dissolve the blockage.

6.9.4 In normal operation nitroprusside gives a yellow background color, which combines with the blue indosalicylate to give an emerald green color. This is the normal color of the solution in the waste.

6.9.5 If baseline drifts, peaks are too wide, or other problems with precision arise, clean the manifold by the following procedure:

1) Place transmission lines in water and pump to clear reagents (2-5 minutes).
2) Place reagent lines in 1 M hydrochloric acid (1 volume of HCl added to 11 volumes of water) and pump for several minutes.
3) Place all transmission lines in water and pump for several minutes.
4) Resume pumping reagents, starting again with the buffer only.

VII. Calibration Standards

7.1 **Standard 1:** Stock Standard 1000 mg NH_3/L

7.1.1 In a 1 L volumetric flask, dissolve 3.819 g ammonium chloride (NH_4Cl) that has been dried for two hours at 110 °C in about 800 mL DI water. Dilute to the mark and invert to mix.

7.2 **Standard 2:** Intermediate Stock Standard 20 mg N/L

7.2.1 To a 1 L volumetric flask, add 5.0 mL of Standard 1 and dilute to the mark with DI water. Invert to mix.

Working Standards (Prepared Daily)	**A**	**B**	**C**	**D**	**E**	**F**	**G**
Concentration in mg/L of N	20.0	10.0	5.00	2.00	1.00	0.50	0.0
Volume of Standard 2 digested and diluted to 100mL with DI water.	Use Std #2 as is	50	25	10	5	2.5	0

VIII. Calculations

8.1 Calibration is done by injecting standards. The data system will then prepare a calibration curve by plotting response versus standard concentration.

8.2 Sample concentration is calculated from the regression equation and reported in mg/L directly from the instrument.

8.3 Report only those values that fall between the lowest and the highest calibration standards. Samples exceeding the highest standard should be diluted and reanalyzed.

8.4 For solids or sediments calculate using the following:

$$\textbf{Total Kjeldahl Nitrogen mg/kg (dry weight)} = \frac{(x)(y)(1000)}{(g)(\%S)}$$

where: x = TKN concentration in sediment digest, mg/L
y = final volume of sediment digest, L
g = wet weight of sample digest, g
%S = percent of solids in sediment sample as a decimal fraction

IX. Sample Collection, Preservation and Storage

9.1 Samples should be preserved to pH <2 with H_2SO_4 and cooled to 4 °C when collected.

9.2 The maximum holding time is 28 days when properly preserved and stored at 4 °C.

9.3 Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water.

9.4 Volume collected should be sufficient to insure a representative sample, allow for replicate analysis (if required), and minimize waste disposal.

9.5 The Federal Register entry which defines standard EPA NPDES and NPDWR methods states that "Manual Distillation is NOT required if comparability data on representative effluent samples are on company file to show that this preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies".

9.5.1 Studies which show that the non-distilled samples give the same recoveries as the manually distilled samples must be documented and updated regularly.

X. Quality Assurance

10.1 The minimum requirements for this method consists of an initial demonstration of laboratory capability, and the analysis of laboratory distilled reagent blanks, fortified blanks and a mid-level CCV in order to evaluate performance.

10.2 Initial Demonstration of Performance

10.2.1 The initial demonstration of performance is used to characterize instrument performance (determination of LCRs and analysis of QCS) and laboratory performance (determination of MDLs) prior to performing analyses by this method.

10.2.2 The linear calibration range (LCR) must be determined initially and verified every six months or whenever a significant change in instrument response is observed or expected.

10.2.2.1 The initial demonstration of linearity must use sufficient standards to insure that the resulting curve is linear.

10.2.2.2 The verification of linearity must use a minimum of a blank and three standards.

10.2.2.3 If any verification data exceeds the initial values by 10%, linearity must be reestablished.

10.2.2.4 If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

10.2.3 Immediately following the calibration, verify the calibration standards and acceptable instrument performance with the preparation and analyses of a quality control sample (QCS).

10.2.3.1 If the determined concentrations are not within 10% of the stated values, performance of the determinative step of the method is unacceptable.

10.2.3.2 The source of the problem must be identified and corrected before either proceeding with the initial determination of MDLs or continuing with on-going analyses.

10.2.4 Method detection limits **(**MDLs) must be established for all analytes, using reagent water (blank) fortified at a concentration of two to three times the estimated instrument detection limit.

10.2.4.1 To determine MDL values, take seven replicate aliquots of the fortified reagent water and process through the entire analytical method.

10.2.4.2 Perform all calculations defined in the method and report the concentration values in the appropriate units.

10.2.4.3 Calculate the MDL as follows:

$$\mathbf{MDL = (t) \times (S)}$$

where: t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom [t = 3.14 for seven replicates]

S = standard deviation of the replicate analyses

10.2.4.4 MDLs should be determined every six months, when a new operator begins work or whenever there is a significant change in the background or instrument response.

10.3 Laboratory Reagent Blank (LRB)

10.3.1 The laboratory must analyze at least one LRB with each batch of 20 samples or less.

10.3.2 Data produced are used to assess contamination from the laboratory environment.

10.3.3 Values that exceed the MDL indicate laboratory or reagent contamination should be suspected and corrective actions must be taken before continuing the analysis.

10.4 Laboratory Fortified Blank (LFB)

10.4.1 Prepare and analyze at least one LFB with each batch of 20 samples or less and calculate accuracy as percent recovery.

10.4.2 If the recovery of any analyte falls outside the required control limits of **90-110%,** that analyte is out of control, and the source of the problem should be identified and resolved before continuing analyses. The LFB analyses data must be used to assess performance against the required control limits of 90-110% or laboratory established control limits.

10.4.3 The control limits must be equal to or better than the required control limits of 90-110%. New control limits can be calculated using the most recent 20-30 data points. This data must be kept on file and be available for review.

10.4.4 At least quarterly, replicates of LFBs should be analyzed to determine the precision of the laboratory measurements. Add these results to the on-going control charts to document data quality.

10.5 Instrument Performance Check Solution (IPC)

10.5.1 For all determinations the IPC (a mid-range check standard) and a calibration blank must be analyzed 1) immediately following daily calibration, 2)after every tenth sample (or more frequently, if required) and 3)at the end of the sample run.

10.5.2 Analysis of the IPC solution and calibration blank immediately following calibration must verify that the instrument is within 10% of calibration. Subsequent analyses of the IPC solution must verify the calibration is still within 10%.

10.5.3 If the calibration cannot be verified within the specified limits, reanalyze the IPC solution. If the second analysis of the IPC solution confirms calibration to be outside the limits, sample analysis must be discontinued, the cause determined and/or in the case of drift, the instrument recalibrated.

10.5.4 All samples following the last acceptable IPC solution must be reanalyzed. The analysis data of the calibration blank and IPC solution must be kept on file with the sample analyses data.

10.6 Laboratory Fortified Sample Matrix (LFM)

10.6.1 The laboratory must add a known amount of analyte to a minimum of 10% of the routine samples. In each case the LFM aliquot must be a duplicate of the aliquot used for sample analysis. The added analyte concentration should be the same as that used in the laboratory fortified blank.

10.6.2 If the concentration of fortification is less than 25% of the background concentration of the matrix the matrix recovery should not be calculated.

10.6.3 Calculate the percent recovery for each analyte, corrected for concentrations measured in the unfortified sample, and compare these values to the designated LFM recovery range 75-125%.

10.6.4 Percent recovery may be calculated using the following equation:

$$\mathbf{R = \frac{C_s - C}{S} \times 100}$$

where: R = percent recovery
C = fortified sample concentration
C_s = sample background concentration
S = concentration equivalent of analyte added to sample

10.6.5 Until sufficient data becomes available (usually a minimum of 20-30 analysis), assess laboratory performance against recovery limits of 75-125%. When sufficient internal performance data becomes available, develop control limits from percent mean recovery.

10.6.6 If the recovery of any analyte falls outside the designated LFM recovery range and the laboratory performance for that analyte is shown to be in control, the recovery problem encountered with the LFM is judged to be either matrix or solution related, not system related.

XI. Reagents and Materials

11.1 Balance—analytical, capable of accurately weighing to the nearest 0.0001g.

11.2 Glassware Class A volumetric flasks and pipettes or plastic containers as required Samples may be stored in plastic or glass.

11.3 Flow injection analysis equipment designed to deliver and react sample and reagents in the required order and ratios, including the following:

a. Autosampler
b. Multichannel proportioning pump
c. Reaction unit or manifold
d. Colorimetric detector
e. Data system
f. Heating Unit
g. Vortex stirrer
h. Use deionized water (10 mega ohm) for all solutions.

11.4 Degassing with helium

11.4.1 To prevent bubble formation, degas the carrier and buffer with helium. Use He at 140 kPa (20 lb/in^2) through a helium degassing tube. Bubble helium through one liter of solution for one minute.

11.4.2 **All reagents used in heated chemistry must be degassed.**

11.5 **Reagent 1 - Mercuric Sulfate Solution**

11.5.1 By Volume: Add approximately 40.0 mL water and 10 mL concentrated sulfuric acid (H_2SO_4) to a 100 mL volumetric flask. Then add 8.0 g red mercuric oxide (HgO). Stir until dissolved, dilute to the mark and invert to mix. Warming the solution while stirring may be required to dissolve the mercuric oxide.

11.6 **Reagent 2 - Digestion Solution**

11.6.1 By Volume: In a 1 L volumetric flask, add 133.0 g potassium sulfate (K_2SO_4) and 200 ml concentrated sulfuric acid (H_2SO_4) to approximately 700 ml water. Add 25.0 mL Reagent 1. Dilute to the mark with water and invert to mix. **Prepare fresh monthly.**

11.7 **Reagent 3 - Buffer**

11.7.1 **Important:** To reduce the possibility of the potassium tartrate being contaminated it is recommended that the tartrate buffer is boiled for 10 minutes. To verify that the tartrate buffer is pure enough compare the reagent baseline to the DI baseline. The baseline, with all reagents flowing should not be greater than 0.15mV difference from just the DI water pumping in all the lines.

11.7.2 By Volume: In a 1 L container add 900 ml water, 50 g potassium tartrate (or potassium sodium tartrate, D, L-$NaKC_4H_4O_6 \cdot 4H_2O$), 50 g sodium hydroxide (NaOH), and 26.8 g sodium phosphate dibasic heptahydrate ($Na_2HPO_4 \cdot 7\ H_2O$) mix until dissolved. Boil for 10 minutes. Cool to room temperature and transfer to a 1 L volumetric flask. Dilute to the mark and invert to mix.

11.8 **Reagent 4 - Sodium Hydroxide (0.8 M)**

11.8.1 By Volume: In a 1 L volumetric flask dissolve 32 g sodium hydroxide (NaOH) in about 800 mL of water. Invert to mix and dilute to the mark.

11.8.2 By Weight: In a 1 L container dissolve 32 g sodium hydroxide (NaOH) in 985g of water.

11.9 **Reagent 5 - Salicylate Nitroprusside**

11.9.1 By Volume: In a 1 L volumetric flask dissolve 150.0 g sodium salicylate [salicylic acid sodium salt, $C_6H_4(OH)(COO)Na$], and 1.0 g sodium nitroprusside [sodium

nitroferricyanide dihydrate, $Na_2Fe(CN)_5NO•2H_2O$] in about 800 mL water. Invert to mix and dilute to the mark. **Store in a dark bottle and prepare fresh monthly.**

11.9.3 By Weight: To a tared 1 L dark container, add 150.0 g sodium salicylate [salicylic acid sodium salt, $C_6H_4(OH)(COO)Na$], 1.0 g sodium nitroprusside [sodium nitroferricyanide dihydrate, $Na_2Fe(CN)_5NO•2H_2O$] and 908g water. Stir or shake until dissolved. **Store in a dark bottle and prepare fresh monthly.**

11.10 **Reagent 6 - Hypochlorite Solution**

11.10.1 By Volume: In a 250 mL volumetric flask, dilute 13.1 mL Regular Clorox Bleach, 6.0% sodium hypochlorite, The Clorox Company, Oakland, CA, (**do not substitute with any other brand of bleach**) to the mark with water (236.9 ml). Invert to mix. **Prepare fresh daily.**

11.10.2 By Weight: To a tared 250 mL container, add 16 g of Regular Clorox Bleach and 234 g DI water. Invert to mix. **Prepare fresh daily.**

11.11 **Reagent 7 - Diluent**

11.11.1 **Important:** Diluent is used to prepare the carrier and for off line dilutions. The sulfuric acid concentration in the carrier needs to match the digestion matrix.

11.11.2 By Volume: In a 2 L volumetric flask, add in the following order: approximately 1800 ml of DI water, 100 mL of concentrated H_2SO_4 and 63.4g of Potassium sulfate (K_2SO_4). Invert to mix and bring to volume. **Prepare fresh weekly.**

XII. Pollution Prevention

12.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice.

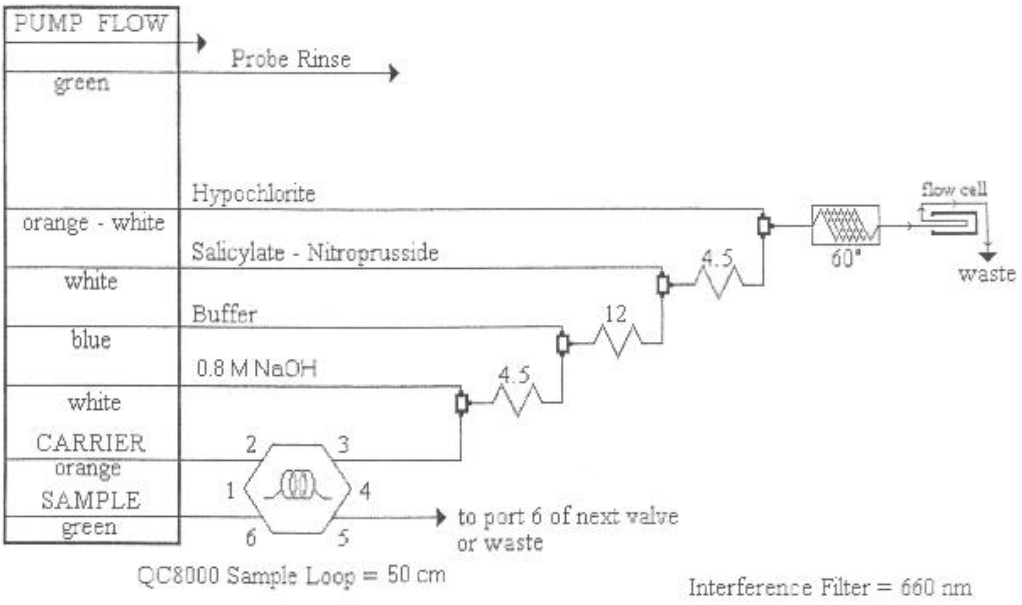
12.3 Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation.

12.4 Quantity of the chemicals purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

XIII. Waste Management

13.1 All waste is handled in accordance with Premier Laboratory's Chemical Hygiene Plan, which is available to all employees and interested parties.

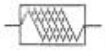
Diagram 1: TKN Manifold Setup



CARRIER is Diluent (reagent 7).

All manifold tubing is **0.8 mm (0.032 in) i.d**. Lachat Part No. 50028. This is **5.2 uL/cm**.

4.5	is	**70**	cm of tubing on a 4.5 cm coil support
12	is	**255**	cm of tubing on a 12 cm coil support

APPARATUS: An injection valve, a 10 mm path length flow cell, and a colorimetric detector module is required. The  indicates 650 cm of tubing wrapped around the heater block at the specified temperature.

SOP Revision History

Date	Previous Revision No.	New Revision No.	Description of Changes	Effective Date	Initiated by
4/14/09	1.0	1.1	Added revision history table Changed the amount of bleach used to make 6% sodium hypochlorite from 15ml to 13.1ml in Section X.	4/14/09	LM
4/17/09	1.1	1.2	Section V: changed 2ml digestion solution to 5ml and changed concentration of 2ppm QC standard to 5ppm	4/17/09	LM
9/8/10	1.2	1.3	Changed format Added Section 5.3	9/22/10	LM

Phosphorus Automated (All Forms)
Method 365.1

Prepared by: Melisa Montgomery, Quality Assurance Officer
Approved by: Robert Stevenson, Laboratory Director
Reviewed and Implemented by: Ronald Warila, General Manager

Reference:

Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1983, Method 365.1

Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998, Method 4500P-F

Technical Report EPA/ CE-81-1, Procedures for Handling and Chemical Analysis of Sediment and Water Samples, May 1981

QuikChem® Method 10-1 15-01-1-A, Lachat Instruments, Milwaukee, WI, August, 2000

I. Applicability

1.1 This method covers the determination of all forms of phosphorus in drinking water, surface water and domestic and industrial wastes. It is also modified to perform soil analysis with a pre-digestion. The applicable range of this method is 0.01 to 2.0 mg/L Phosphate as P.

II. Important Notes

2.1 Sample containers may be of plastic material, such as a cubitainer, or of Pyrex glass.

2.2 If the analysis cannot be performed the day of collection, the sample should be preserved by the addition of 2 mL of conc. H_2SO_4 per liter and refrigeration at 4°C.

2.3 Ortho phosphate is **never preserved**.

2.4 Concentrations of ferric iron (Fe^{3}-) greater than 50 mg/L will cause a negative error due to precipitation of, and subsequent loss, of orthophosphate. Samples high in iron can be pretreated with sodium bisulfite to eliminate this interference. Treatment with bisulfite will also remove the interference due to arsenates.

2.5 Glassware contamination is a problem in low level phosphorus determinations. Glassware should be washed with 1:1 HC1 and rinsed with deionized water. Commercial detergents

should rarely be needed but, if they are used, use special phosphate-free preparations for lab glassware.

2.6 All quality control samples are digested.

2.7 All glassware is cleaned with 1:1 HCl.

III. Definitions

3.1 Calibration Blank (CB) – A volume of reagent water in the same matrix as the calibration standards, but without the analyte.

3.2 Calibration Standard (CAL) – A solution prepared from the primary dilution standard solution or stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

3.3 Instrument Performance Check Solution (IPC) – A solution of one or more method analytes used to evaluate the performance of the instrument system with respect to a defined set of criteria.

3.4 Laboratory Fortified Blank (LFB) – An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The LSB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

3.5 Laboratory Fortified Matrix (LFM) – An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LSM is analyzed exactly like sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.

3.6 Laboratory Reagent Blank (LRB) – An aliquot of reagent water or other blank matrices that is digested exactly as a sample in including exposure to all glassware, equipment, and reagents that are used with other samples. The LRB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents or the apparatus.

3.7 Linear Calibration Range (LCR) – The concentration range over which the instrument response is linear.

3.8 Method Detection Limit (MDL) – The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

3.9 Quality Control Sample (QCS) – A solution of method analytes of known concentrations that is used to spike an aliquot of LRB or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

3.10 Stock Standard Solution (SSS) – A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

IV. Procedure for Ortho Phosphorus

4.1 Analyze unfiltered, with no digestion or hydrolysis. Holding time is 48 hours with no preservative. Proceed to calibration section.

V. Procedure for Total Phosphorus

5.1 Digestion of Aqueous samples

5.1.1 To 50 mL of sample, add 1 drop phenolphthalein indicator solution. If a red color appears, add H_2SO_4 solution dropwise until color is discharged.

Then add 1 mL H_2SO_4 solution to all samples, blanks and QC samples.

5.1.2 Add 0.4 g of ammonium persulfate.

5.1.3 Boil gently on a preheated hot plate for approximately 30-40 minutes or until a final volume of about 10 mL is reached, or if grey smoke fills the flask. Do not allow sample to go to dryness. Redigest if sample goes to dryness.

5.1.4 Cool sample and dilute to approximately 30 mL with distilled water. Add 1 mL 6N NaOH then dilute to a final volume of 50mL.

5.1.5 If samples are not clear at this point, filter the sample and an aliquot of both the LFB and LRB to be run as filter QC samples.

5.2 Procedures for Sediment Samples

5.2.1 Persulfate digestion

5.2.1.1 Weigh 0.5-1.0g dry weight equivalent of the sample and transfer to a 150-mL beaker.

5.2.1.2 Add 10 mL 30 percent H_2SO_4 and 2 g potassium persulfate.

5.2.1.3 Mix the suspension and heat on a hot plate for 1 hr.

5.2.1.4 Filter with a pre-rinsed paper filter (Watman 41 or equivalent) into a l00-mL volumetric flask and dilute to volume.

5.2.1.5 Prepare a separate LFB, LRB and LFM/LFMD for sediments.

VI. Calculations

6.1 Report only the values that are less than 90% of the highest standard in the calibration. Dilute appropriately and re-analyze samples that do not meet this criteria

6.2 Aqueous Samples

6.2.1 Direct reading in mg/L from the Lachat

6.3 Solid Samples

6.3.1 Calculate the phosphate concentration on a dry weight basis as follows:

$$\textbf{Total phosphate mg/kg (dry weight)} = \frac{(x)(y)(1000)}{(g)(\%S)}$$

where: x = phosphate concentration in sediment digest, **mg/L**
y = final volume of sediment digest, **L**
g = wet weight of sample digest, **g**
%S = percent of solids in sediment sample, **as a decimal fraction**

VII. Standards and Reagent Preparation

7.1 Preparation of Reagents

7.1.1 Use deionized water for all solutions.

7.1.2 Degassing with helium

7.1.2.1 To prevent bubble formation, degas the carrier solution with helium. Use He at 5-20 psi through a disposable narrow tip pipette. Bubble He vigorously through the solution for one minute. Dispose of the pipette after each use.

7.1.3 **Reagent 1 – Stock Ammonium Molybdate Solution**

7.1.3.1 In a 1 L volumetric flask dissolve 40.0 g ammonium molybdate tetrahydrate [$(NH_4)_6Mo_7O_{24} \cdot 4H_2O$] in approximately 800 mL DI water.

7.1.3.2 Dilute to the mark and stir for four hours.

7.1.3.3 Store in plastic and refrigerate.

7.1.3.4 May be stored up to two months when kept refrigerated.

7.1.4 **Reagent 2 – Stock Antimony Potassium Tartrate Solution**

7.1.4.1 In a 1 L volumetric flask, dissolve 3.0 g antimony potassium tartrate (potassium antimony tartrate hemihydrate $K(SbO)C_4H_4O_6 \cdot \frac{1}{2}H_2O$) or dissolve 3.22 g antimony potassium tartrate (potassium antimony tartrate trihydrate $K_2(C_4H_2O_6Sb)_2 \cdot 3H_2O$) in approximately 800 mL of DI water.

7.1.4.2 Dilute to the mark and invert three times.

7.1.4.3 Store in a dark bottle and refrigerate.

7.1.4.4 Maybe stored up to two months when kept refrigerated.

7.1.5 **Reagent 3 – Molybdate Color Reagent**

7.1.5.1 To a 1 L volumetric flask add about 500 mL DI water.

7.1.5.2 Add 35.0 mL concentrated sulfuric acid and swirl to mix.

(CAUTION: The reaction is exothermic; it will get warm!)

7.1.5.3 When it can be comfortably handled, add 72.0 mL Stock Antimony Potassium Tartrate Solution (Reagent 2) and 213 mL Stock Ammonium Molybdate Solution (Reagent 1).

7.1.5.4 Dilute to the mark and invert three times.

7.1.5.5 Degas with helium.

7.1.5.6 Prepare fresh weekly.

7.1.6 **Reagent 4 – Ascorbic Acid Reducing Solution, 0.33 M**

7.1.6.1 In a 1 L volumetric flask dissolve 60.0 g granular ascorbic acid in about 700 mL of DI water.

7.1.6.2 Dilute to the mark and invert to mix.

7.1.6.3 Add 1.0 g dodecyl sulfate ($CH_3(CH_2)_{11}OSO_3Na$).

7.1.6.4 Prepare fresh weekly.

7.1.6.5 Discard if the solution becomes yellow.

7.1.7 **Reagent 5 – Sodium Hydroxide - EDTA Rinse**

7.1.7.1 Dissolve 65 g sodium hydroxide (NaOH) and 6 g tetrasodium ethylenediamine tetraacetic acid (Na_4EDTA) in 1.0 L DI water.

7.1.8 **Reagent 6 – Sulfuric Acid Solution, 11 N**

7.1.8.1 Carefully add 300mL concentrated H_2SO_4 to approximately 600mL of distilled water and dilute to 1L with distilled water.

7.2 Preparation of Standards

7.2.1 **Stock Standard Solution #1: 250.0 mg/L of Phosphate as P**

7.2.1.1 In a 500 mL volumetric flask dissolve 0.5495 g primary standard grade anhydrous potassium phosphate monobasic (KH_2PO_4) that has been dried for one hour at 105 ^oC in approximately 400 mL water.

7.2.1.2 Dilute to the mark with DI water and invert to mix.

7.2.2 **Stock Standard Solution #2: 50.0 mg/L of Phosphate as P**

7.2.2.1 In a 200 mL volumetric flask, dilute 40.0 mL Stock Standard Solution #1 to the mark with DI water.

7.2.2.2 Invert to mix.

7.3 Working Standards

7.3.1 Prepare fresh daily using deionized H_2O as shown below:

Standard	**A**	**B**	**C**	**D**	**E**	**F**	**G**	**Blank**
Concentration, mg/L	2	1	0.5	0.2	0.05	0.02	0.01	--
mL of Solution #2	10	5	2.5	1.0	0.25	0.1	--	--
mL of Standard A	--	--	--	--	--	--	1	--
Final Volume, mL	250 mL							

VIII. Instrumental Analysis

8.1 pH Adjustment of Samples

8.1.1 Test the pH of all samples submitted for orthophosphate analysis using the pH test strip method.

8.1.2 If samples have a pH >8, add 1 drop of phenolphthalein indicator to a 50 mL aliquot of sample. If a red color develops, add 11 N sulfuric acid (310 mL concentrated H_2SO_4/L) drop-wise to just discharge the color. Acidic samples (pH<4) must be neutralized with 1 N NaOH (40 g NaOH/L).

8.2 Prepare reagent and standards as described.

8.3 Set up manifold as shown in Diagram 1.

8.4 Pump DI water through all reagent lines and check for leaks and smooth flow. Switch to reagents and allow the system to equilibrate until a stable baseline is achieved.

8.5 Prime the auto diluter pump with the carrier reagent.

8.6 Input the sample data into the sample tray application.

8.7 Calibrate using the prepared standards to create a curve with a correlation coefficient of 0.995 or better.

8.8 Analyze the samples and QC in the established sequence.

IX. Quality Assurance

9.1 The minimum requirements for this method consists of an initial demonstration of laboratory capability, and the analysis of laboratory distilled reagent blanks, fortified blanks and a mid-level CCV in order to evaluate performance. Undigested reagent blanks, fortified blanks and mid level CCV may be used when digestion of the analyzed samples in not required.

9.2 Initial Demonstration of Performance

9.2.1 The initial demonstration of performance is used to characterize instrument performance (determination of LCRs and analysis of QCS) and laboratory performance (determination of MDLs) prior to performing analyses by this method.

9.2.2 Linear Calibration Range (LCR)

9.2.2.1 The LCR must be determined initially and verified every six months or whenever a significant change in instrument response is observed or expected.

9.2.2.2 The initial demonstration of linearity must use sufficient standards to insure that the resulting curve is linear.

9.2.2.3 The verification of linearity must use a minimum of a blank and three standards. If any verification data exceeds the initial values by 10%, linearity must be reestablished. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

9.2.3 Quality Control Sample (QCS)

9.2.3.1 Immediately following the calibration, verify the calibration standards and acceptable instrument performance with the preparation and analyses of a QCS.

9.2.3.2 If the determined concentrations are not within 10% of the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before either proceeding with the initial determination of MDLs or continuing with on-going analyses.

9.2.4 Method Detection Limit (MDL)

9.2.4.1 MDLs must be established for all analytes, using reagent water (blank) fortified at a concentration of two to three times the estimated instrument detection limit.

9.2.4.2 To determine MDL values, take seven replicate aliquots of the fortified reagent water and process through the entire analytical method. Perform all calculations defined in the method and report the concentration values in the appropriate units.

9.2.4.3 Calculate the MDL as follows:

$$\mathbf{MDL = (t) \times (S)}$$

where: t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom [t= 3.14 for seven replicates]
S = standard deviation of the replicate analyses

9.2.4.4 MDLs should be determined every year, when a new operator begins work or whenever there is a significant change in the background or instrument response.

9.3 Laboratory Reagent Blank (LRB)

9.3.1 The laboratory must analyze at least one LRB with each batch of 20 samples or less.

9.3.2 Data produced are used to assess contamination from the laboratory environment.

9.3.3 Values that exceed the MDL indicate laboratory or reagent contamination should be suspected and corrective actions must be taken before continuing the analysis.

9.4 Laboratory Fortified Blank (LFB)

9.4.1 At least one LFB must be analyzed with each batch of 20 samples or less.

9.4.2 Calculate accuracy as percent recovery. If the recovery of any analyte falls outside the required control limits of 90-110%, that analyte is judged out of control, and the source of the problem should be identified and resolved before continuing analyses. The LFB analyses data must be used to assess performance against the required control limits of 90-110% or laboratory established control limits.

9.4.2.1 The control limits must be equal to or better than the required control limits of 90-110%. New control limits can be calculated using the most recent 20-30 data points. This data must be kept on file and be available for review.

9.4.3 Prepare a 1.25 ppm LFB by adding 0.5 mL of 250 ppm phosphate stock solution to 100 mL of distilled water. Digest with the sample batch for total phosphate.

9.4.4 An orthophosphate LFB is an aliquot of the 1.0 ppm standard that has been diluted from the stock with DI water.

9.4.5 At least quarterly, replicates of LFBs should be analyzed to determine the precision of the laboratory measurements. Add these results to the on-going control charts to document data quality.

9.5 Instrument Performance Check Solution (IPC)

9.5.1 For all determinations the IPC (a mid-range check standard) must be analyzed and a calibration blank immediately following daily calibration, and after every tenth sample (or more frequently, if required) and at the end of the sample run.

9.5.2 Analysis of the IPC solution and calibration blank immediately following calibration must verify that the instrument is within 10% of calibration. Subsequent analyses of the IPC solution must verify the calibration is still within 10%.

9.5.3 If the calibration cannot be verified within the specified limits, reanalyze the IPC solution.

9.5.4 If the second analysis of the IPC solution confirms calibration to be outside the limits, sample analysis must be discontinued, the cause determined and/or in the case of drift, the instrument recalibrated.

9.5.5 All samples following the last acceptable IPC solution must be reanalyzed.

9.5.6 The analysis data of the calibration blank and IPC solution must be kept on file with the sample analyses data.

9.6 Laboratory Fortified Sample Matrix (LFM)

9.6.1 The laboratory must add a known amount of analyte to a minimum of 10% of the routine samples.

9.6.2 In each case the LFM aliquot must be a duplicate of the aliquot used for sample analysis.

9.6.3 The added analyte concentration should be the same as that used in the laboratory fortified blank.

9.6.4 For total phosphate, the LFM undergoes the digestion process.

9.6.5 The LFM is prepared by adding 0.5 mL of 250 ppm stock to 100 mL of sample. This will result in a 1.25-ppm spike.

9.6.6 If orthophosphate is needed, add 0.1 mL of 250-ppm stock to 25 mL of sample. This will result in a 1.0-ppm spike. The orthophosphate LFM is not digested.

9.6.7 If the concentration of fortification is less than 25% of the background concentration of the matrix the matrix recovery should not be calculated.

9.6.8 Calculate the percent recovery for each analyte, corrected for concentrations measured in the unfortified sample, and compare these values to the designated LFM recovery range 75-125%. Percent recovery may be calculated using the following equation:

$$R = \frac{C_s - C}{s} \times 100$$

where: R = percent recovery
C = fortified sample concentration
C_s = sample background concentration
s = concentration equivalent of analyte added to sample

9.6.9 Until sufficient data becomes available (usually a minimum of 20-30 analysis), assess laboratory performance against recovery limits of 75-125%.

9.6.10 When sufficient internal performance data becomes available, develop control limits from percent mean recovery. If the recovery of any analyte falls outside the designated LFM recovery range and the laboratory performance for that analyte is shown to be in control, the recovery problem encountered with the LFM is judged to be either matrix or solution related, not system related.

X. Pollution Prevention

10.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in the laboratory. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice.

10.2 Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation by the following means:

10.2.1 Insure that the quantity of the chemicals purchased is based on expected usage during its shelf life and the disposal cost of unused material.

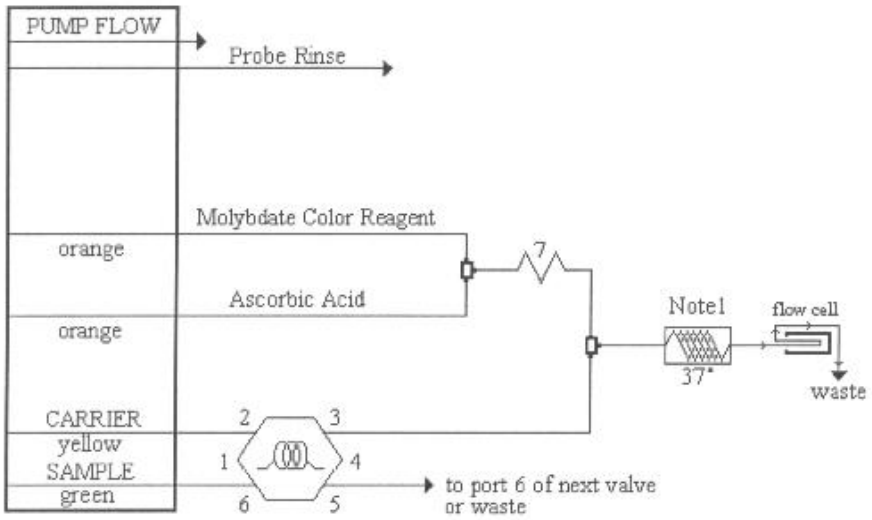
10.2.2 Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

10.2.3 Control the usage by closely monitoring the instrument operation to avoid pumping reagents through after sample run has completed.

XI. Waste Management

11.1 All waste is handled in accordance with Premier Laboratory's Chemical Hygiene Plan, which is mandatory reading for all employees and is readily available for any interested parties.

Diagram 1: Phosphate Manifold Setup



Carrier: DI water
Manifold Tubing: 0.8 mm (0.032 in) i.d. This is 5.2 µL/cm.
AE Sample Loop: 70 cm x 0.8 mm i.d.
QC8000 Sample Loop: 75.5 cm x 0.8 mm i.d.
Interference Filter: 880 nm

Apparatus: An injection valve, a 10 mm path length flow cell , and a colorimetric detector module is required. The  shows 175 cm of tubing wrapped around the heater block at 37°C.

7: 135 cm of tubing on a 7 cm coil support

Note 1: 175 cm of tubing on the heater.

SOP Revision History

Date	Previous Revision No.	New Revision No.	Description of Changes	Effective Date	Initiated by
6/8/09	2.2	2.3	Added revision history table	6/8/09	LM
2/22/11	2.3	2.4	Clarified addition of H_2SO_4 in Section 5.1.1 Clarified filtration requirement in Section 5.1.5 Added requirement to prepare LFB, LRB, and LFM/LFMD for sediments in Section 5.2.1.5 Changed standards preparation instructions for smaller amounts in Section 7.2	2/25/11	BS, LM

Total Solids (% Solids)
SM 2540 G

Prepared by: Melisa Montgomery, Quality Assurance Officer
Approved by: Robert Stevenson, Laboratory Director
Reviewed and Implemented by: Ronald Warila, General Manager

Reference:

Standard Methods, 19th Edition, 1995, Method 2540G

I. Applicability

1.1 Analyte: % solids

1.2 Matrix: Soil, sludge, solids

1.3 Regulation: None

II. Important Notes

2.1 A high amount of volatile matter in the sample could lead to lower results.

III. Procedure

3.1 Dry a weighing dish for at least one hour at 103 - 105 °C. Cool and store in a desiccator until use.

3.2 Weigh a dried weighing dish and record the weight to the nearest 0.01 g.

3.3 Transfer 10 g of sample to the drying dish and record the weight of the dish plus the sample to the nearest 0.01 g.

3.4 Place the weighing dish with the sample in a drying oven at 103 - 105 °C. Dry the sample overnight. In cases where the results are needed with an accelerated turn around, the samples may be dried initially for 4 hours. However, the **Drying Efficiency Check** (see below) must be performed on all samples run until the criterion is met for each sample.

3.5 Cool the weighing dish in a desiccator. Weigh the dish with sample and record the weight to the nearest 0.01g.

IV. Calculations

$$S = \frac{100 \times (F - D)}{I - D}$$

where: S = % solids
D = weight of empty weighing dish, g
I = initial weight of weighing dish plus sample before drying, g
F = final weight of weighing dish plus sample after drying, g

Report results to 3 significant figures in the LIMS system.

V. Quality Assurance

5.1 Drying Efficiency Check

5.1.1 Select one representative sample from a batch of 20 samples or less.

5.1.2 Repeat drying (1hr), cooling/desiccating, and weighing step.

5.1.3 Reweigh and calculate the change in weight.

5.1.4 The weight change should be less than 4% or 50 mg, whichever is less, of the initial dry weight determined

5.1.5 If the weight change exceeds the above limit, return all samples to the drying oven for 2 hrs, cool/desiccate, and weigh.

5.1.6 Repeat Drying Efficiency Check on a different representative sample until the passing criteria is met.

5.2 Sample Duplicate

5.2.1 Analyze sample duplicates at a minimum frequency of one per 20 samples or one per month, whichever is more frequent. The %RPD must be less than or equal to ± 20%.

5.3 Analysis Blank

5.3.1 Analyze a blank with each batch of samples analyzed. The result for the blank must be less than the quantitation limit.

VI. Reagents and Materials

6.1 Disposable weighing dishes: Purchased

6.2 Weighing balance, capable of weighing to 10 mg

6.3 Stainless steel spatula or equivalent

VII. Safety

7.1 Every sample should be considered a hazardous when performing the analysis.

7.2 Standard laboratory safety guidelines must be adhered to.

7.3 Gloves, eye protection and lab coats must be worn during sample retrieval, analysis and disposal.

VIII. Pollution Prevention

8.1 Any and all remaining unused sample must be returned to the 4°C storage, sealed tightly in the original container.

8.2 Benches and surrounding surfaces must be cleaned and wiped dry with paper towels.

IX. Waste management

9.1 Analyzed samples and used disposable equipment must be collected and disposed of in a manner consistent with the Premier Laboratory Chemical Hygiene Plan.

X. Method Performance

10.1 Performance data is not currently available.

SOP Revision History

Date	Previous Revision No.	New Revision No.	Description of Changes	Effective Date	Initiated by
6/30/09	2.2	2.3	Added revision history table	7/1/09	LM
9/22/10	2.3	2.4	Changed format	9/22/10	LM

GeoTesting EXPRESS

Standard Operating Procedure ASTM D 422

Technical Information

Reference Number:	ASTM D 422-latest revision
Test Method Title:	Test Method for Particle Size Analysis of Soils
Test Property:	Grain Size Analysis
Test Specimen Size:	Passing #10 sieve: 115 g sandy soils, 65 g silty or clayey soils Retained on #10 sieve: see test standard (based on largest particle size)
Number of Test Specimens:	1 representative sample obtained by quartering, mixing or splitting
Test Equipment:	Hydrometer (ASTM) Sedimentation Cylinder Stirring Apparatus (blender) Dispersion Cup Drying containers Balance readable to 0.01 gram for material passing #10 sieve or 0.1% of mass for material retained on #10 sieve Thermometer readable to 0.5 ºC Various sieves 250 mL beaker Drying oven capable of maintaining a temperature of 110 ± 5 ºC Dispersing agent mixture (40 g/L of Sodium Hexametaphosphate solution) Mechanical sieve shaker Distilled Water Spray Bottle Wash pan

Standard Operating Procedure

Sampling

1. Collect a representative sample and perform a moisture content test in accordance with ASTM D 2216.

2. Collect another representative sample to be used for the particle size analysis. Base specimen size on test standard (based on largest particle size). Record specimen wet weight.

Splitting / Washing sample on #200 sieve

3. Add 125 ml of dispersing agent into sample container. Stir well and allow to soak for at least 16 hours.

4. Rinse sample into dispersion cup and use stirring apparatus (blender) to further disperse sample for 1 minute.

5. Wash the test specimen from the dispersion cup, using distilled water, over the No. 200 sieve into a container. Be sure to collect all washings in the container. Use only 800 ml of distilled water for the washing operation.

6. Transfer the portion retained on the No. 200 into a tare and place in a drying oven.

7. Wash the minus No. 200 sieve material into a Sedimentation cylinder.

Sieve analysis of portion retained on #200 sieve

8. Separate the portion retained on #200 sieve into a series of fractions using various sieve sizes ranging from 3 inch to #200. Set up in mechanical shaker and shake for 10 minutes. Determine the mass retained on each sieve by weighing and recording mass to nearest 0.1 % of sample mass.

Hydrometer analysis of portion passing #200 sieve

9. Add distilled water to the1000 mL point. Place a rubber stopper over the open end and turn the cylinder upside down and back for a period of 1 minute (should be 60 turns per minute). Set the cylinder down, remove stopper and wash any adhering soil into the cylinder. Begin to take and record hydrometer readings at the following intervals: 2, 5, 15, 30, 60,120, 240 and approximately 1440 minutes. After each reading, the temperature of the solution should be recorded.

10. Calculations: Use initial moisture content and initial wet weight of test specimen to calculate initial dry weight of test specimen. Use reporting software to enter data and calculate % passing and retained for each sieve size and hydrometer readings.

11. Report: sample identification, sample description, percentage passing or retained on each sieve fraction (tabular and graphical).

Appendix B

Sediment Core Photographic Log

Photograph No.: 1
SC1A-A

Photograph No.: 2
SC1A-B

ESS Filepath: J:\W301-000 Weymouth Whitmans Pond\Technical\Photos\Sediment-coring-photolog.ppt

Photograph No.: 3
SC1A-C

Photograph No.: 4
SC1B-A

ESS Filepath: J:\W301-000 Weymouth Whitmans Pond\Technical\Photos\Sediment-coring-photolog.ppt

Photograph No.: 5
SC1B-B

Photograph No.: 6
SC1C-A

ESS Filepath: J:\W301-000 Weymouth Whitmans Pond\Technical\Photos\Sediment-coring-photolog.ppt

Photograph No.: 7
SC1C-B

Photograph No.: 8
SC2A-A

ESS Filepath: J:\W301-000 Weymouth Whitmans Pond\Technical\Photos\Sediment-coring-photolog.ppt

Photograph No.: 9
SC2B-A

Photograph No.: 10
SC2B-B

ESS Filepath: J:\W301-000 Weymouth Whitmans Pond\Technical\Photos\Sediment-coring-photolog.ppt

Photograph No.: 11
SC2C-A

Photograph No.: 12
SC2C-B

ESS Filepath: J:\W301-000 Weymouth Whitmans Pond\Technical\Photos\Sediment-coring-photolog.ppt

Photograph No.: 13
SC2C-C

Photograph No.: 14
SC3C-A

ESS Filepath: J:\W301-000 Weymouth Whitmans Pond\Technical\Photos\Sediment-coring-photolog.ppt

Photograph No.: 15
SC3C-B

Photograph No.: 16
SC3B-A

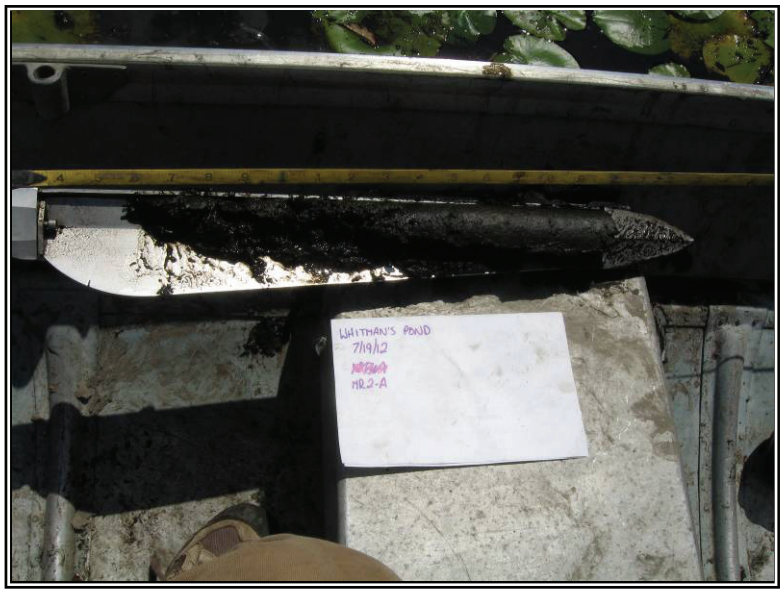
ESS Filepath: J:\W301-000 Weymouth Whitmans Pond\Technical\Photos\Sediment-coring-photolog.ppt

Photograph No.: 17
SC3B-B

Photograph No.: 18
SC3A-A

ESS Filepath: J:\W301-000 Weymouth Whitmans Pond\Technical\Photos\Sediment-coring-photolog.ppt

Photograph No.: 19
MR3-A



Photograph No.: 20
MR2-A

ESS Filepath: J:\W301-000 Weymouth Whitmans Pond\Technical\Photos\Sediment-coring-photolog.ppt

Photograph No.: 21
MR2-B

Photograph No.: 22
MR1-A

ESS Filepath: J:\W301-000 Weymouth Whitmans Pond\Technical\Photos\Sediment-coring-photolog.ppt

Photograph No.: 23
MB3-A

Photograph No.: 24
MB3-B

ESS Filepath: J:\W301-000 Weymouth Whitmans Pond\Technical\Photos\Sediment-coring-photolog.ppt

Photograph No.: 25
MB1-A

Photograph No.: 26
MB1-B

ESS Filepath: J:\W301-000 Weymouth Whitmans Pond\Technical\Photos\Sediment-coring-photolog.ppt

Photograph No.: 27
MB1-C

Photograph No.: 28
MB2-A

ESS Filepath: J:\W301-000 Weymouth Whitmans Pond\Technical\Photos\Sediment-coring-photolog.ppt

Photograph No.: 29
MB2-B

Photograph No.: 30
MB2-C

ESS Filepath: J:\W301-000 Weymouth Whitmans Pond\Technical\Photos\Sediment-coring-photolog.ppt

Photograph No.: 31
WC3-A

Photograph No.: 32
WC3-B

ESS Filepath: J:\W301-000 Weymouth Whitmans Pond\Technical\Photos\Sediment-coring-photolog.ppt

Photograph No.: 33
WC3-C

Photograph No.: 34
WC2-A

ESS Filepath: J:\W301-000 Weymouth Whitmans Pond\Technical\Photos\Sediment-coring-photolog.ppt

Photograph No.: 35
WC2-B

Photograph No.: 36
WC2-C

ESS Filepath: J:\W301-000 Weymouth Whitmans Pond\Technical\Photos\Sediment-coring-photolog.ppt

Photograph No.: 37
WC1-A

Photograph No.: 38
WC1-B

ESS Filepath: J:\W301-000 Weymouth Whitmans Pond\Technical\Photos\Sediment-coring-photolog.ppt

Photograph No.: 39
WC1-C

ESS Filepath: J:\W301-000 Weymouth Whitmans Pond\Technical\Photos\Sediment-coring-photolog.ppt

Appendix C

Hydrologic Assessment Sheet

Whitman's Pond - HYDROLOGIC ASSESSMENT

								Source:
Watershed for Whitman's Pond =		8,142.0	acres		354,665,520	SF	13 mi2	ESS delineation based on MassGIS USGS topos
Pond Area		186.5	acres		8,123,940	SF	754,739 meters2	ESS, 2012, calculation in GIS
Area of Watershed - Pond Area		7,955.5	acres		346,541,580	SF		Calculation
Lake Circumference		32,724.0	feet					ESS, 2012, calculation in GIS
Lake Volume		71,441,565.4	cubic feet				2,023,000.1 meters3	ESS, 2012, calculation based on GIS and field data
Area influenced by seepage		163,620.0	ft2	=	15,201	m2		ESS estimate derived from pond circumference
Groundwater (data)		2	l/m2/day	=	0.071	cf/m2/day		ESS estimate based on unpublished data
				=	1,073.2	cf/day		Calculation
				=	0.012	cfs		Calculation
Annual PPT/yr		42.53	inches					Logan Airport 30 year record
Annual PPT - ET	28.50	2.37	ft/yr		0.61	cfs		Precip on pond minus regional ET (NRCC, 2012)
Runoff (watershed)	14.03	1.17	ft/yr		12.85	cfs		Calculation
Base Flow (Streams) as measured during dry weather - Average =					8.40	cfs		Sum of primary tributary estimates (note: StreamStats D60 for outlet is 8.98)
Old Swamp River		4.15						USGS Gage at Rte 3/Stream Stats, D60 Weighted Estimate Flow
Mill River		4.25						StreamStats, D60 Flow, modeled at Mill River mouth in Whitman's Pond

	Ground	PPT	Surfacewater	Total	
Dry	0.012	0.000	8.400	**8.412**	
Wet	0.000	0.612	12.852	**13.464**	
Total	**0.012**	**0.612**	**21.252**	**21.876**	cfs

Estimated range of total annual input into lake:
(1.5 to 2 cfs/sq mi of watershed) =
19.08 to 25.44 cfs

Appendix D

Sediment Quality Results

Sediment Analysis Results from Whitman's Pond, Weymouth, MA.

Analyte	SC1-Comp	SC2-Comp	SC3-Comp	MR-Comp	MB-Comp	WC-Comp	MCP[1]	BUD[2]	Lined Landfill[3]
Moisture Content (%)	88	78	87	77	91	89	NR	NR	NR
Total Organic Carbon in Soil (mg/kg)	186000	96900	16700	59000	226000	280000	NR	NR	NR
Mercury by SW-846 7471 in SW (mg/kg):									
Mercury	*0.17*	*0.089*	*0.15*	1.2	*0.23*	*0.18*	20	8.7	10
Trace Metals by 6010B (mg/kg):									
Arsenic	*4.2*	*2.2*	*3.8*	20	*5.8*	4.9	20	11	40
Cadmium	*0.85*	0.55	1.7	8.1	1.3	0.99	2	0.8	30
Chromium	11	17	38	38	10	5.4	30	11	1000
Copper	12	9.7	20	59	24	20	1000	NR	NR
Lead	14	20	58	400	39	7.9	300	19	2000
Nickel	7.2	5.2	45	22	9.1	4.2	20	7.2	NR
Zinc	63	28	93	610	92	19	2500	28	NR
Volatiles by 8260B (GA/GW-1/S-1) (ug/kg):									
Acetone	*14000*	*9500*	1600	1000	*10000*	8000	6000	330	NR
Acrylonitrile	*36*	*24*	*33*	*18*	*100*	*24*	NR	NR	NR
Benzene	*36*	*24*	*33*	24	*100*	*24*	2000	150	NR
Bromobenzene	*36*	*24*	*33*	*18*	*100*	*24*	NR	NR	NR
Bromochloromethane	*36*	*24*	*33*	*18*	*100*	*24*	NR	NR	NR
Bromodichloromethane	*36*	*24*	*33*	*18*	*100*	*24*	100	5	NR
Bromoform	*36*	*24*	*33*	*18*	*100*	*24*	100	7	NR
Bromomethane	*36*	*24*	*33*	*18*	*100*	*24*	500	10	NR
2-Butanone (MEK)	*3600*	*2400*	270	240	1500	410	NR	NR	NR
n-Butylbenzene	*36*	*24*	*33*	*18*	*100*	*24*	NR	NR	NR
sec-Butylbenzene	*36*	*24*	*33*	*18*	*100*	*24*	NR	NR	NR
tert-Butylbenzene	*36*	*24*	*33*	*18*	*100*	*24*	NR	NR	NR
Carbon disulfide	43	*24*	*33*	*18*	160	*24*	NR	NR	NR
Carbon tetrachloride	*36*	*24*	*33*	*18*	*100*	*24*	10000	3900	NR
Chlorobenzene	*36*	*24*	*33*	*18*	*100*	*24*	1000	28	NR
Chloroethane	*36*	*24*	*33*	*18*	*100*	*24*	NR	NR	NR
Chloroform	*36*	*24*	*33*	*18*	*100*	*24*	400	5	NR
Chloromethane	*36*	*24*	*33*	*18*	*100*	*24*	NR	NR	NR
2-Chlorotoluene	*36*	*24*	*33*	*18*	*100*	*24*	NR	NR	NR
4-Chlorotoluene	*36*	*24*	*33*	*18*	*100*	*24*	NR	NR	NR
1,2-Dibromo-3-chloropropane (DBCP)	*36*	*24*	*33*	*18*	*100*	*24*	NR	NR	NR
Dibromochloromethane	*36*	*24*	*33*	*18*	*100*	*24*	5	5	NR
1,2-Dibromoethane (EDB)	*36*	*24*	*33*	*18*	*100*	*24*	NR	NR	NR
Dibromomethane	*36*	*24*	*33*	*18*	*100*	*24*	NR	NR	NR
1,2-Dichlorobenzene	*36*	*24*	*33*	*18*	*100*	*24*	9000	NR	NR
1,3-Dichlorobenzene	*36*	*24*	*33*	*18*	*100*	*24*	1000	NR	NR
1,4-Dichlorobenzene	*36*	*24*	*33*	*18*	*100*	*24*	700	NR	NR
Dichlorodifluoromethane	*36*	*24*	*33*	*18*	*100*	*24*	NR	NR	NR
1,1-Dichloroethane	*36*	*24*	*33*	*18*	*100*	*24*	400	200	NR
1,2-Dichloroethane	*36*	*24*	*33*	*18*	*100*	*24*	100	5	NR
1,1-Dichloroethene	*36*	*24*	*33*	*18*	*100*	*24*	3	NR	NR
cis-1,2-Dichloroethene	*36*	*24*	*33*	*18*	*100*	*24*	300	13	NR
trans-1,2-Dichloroethene	*36*	*24*	*33*	*18*	*100*	*24*	100	92	NR
1,2-Dichloropropane	*36*	*24*	*33*	*18*	*100*	*24*	100	5	NR
1,3-Dichloropropane	*36*	*24*	*33*	*18*	*100*	*24*	NR	NR	NR
2,2-Dichloropropane	*36*	*24*	*33*	*18*	*100*	*24*	NR	NR	NR
1,1-Dichloropropene	*36*	*24*	*33*	*18*	*100*	*24*	NR	NR	NR
cis-1,3-Dichloropropene	*36*	*24*	*33*	*18*	*100*	*24*	10	NR	NR
trans-1,3-Dichloropropene	*36*	*24*	*33*	*18*	*100*	*24*	10	19	NR
Diethyl ether	*36*	*24*	*33*	*18*	*100*	*24*	NR	NR	NR
1,4-Dioxane	*140*	*95*	*130*	*71*	*420*	*96*	2000	NR	NR
Ethylbenzene	*36*	*24*	*33*	*18*	*100*	*24*	40000	1900	NR
Hexachlorobutadiene	*36*	*24*	*33*	*18*	*100*	*24*	6000	3000	NR
2-Hexanone	*73*	*47*	*67*	*36*	*210*	*48*	NR	NR	NR
Isopropylbenzene	*36*	*24*	*33*	*18*	*100*	*24*	NR	NR	NR
4-Isopropyltoluene	*36*	*24*	*33*	*18*	*100*	*24*	NR	NR	NR
Methyl tert-butyl ether (MTBE)	*36*	*24*	*33*	*18*	*100*	*24*	1000	140	NR
4-Methyl-2-pentanone (MIBK)	*73*	*47*	*67*	*36*	*210*	*48*	NR	NR	NR
Methylene chloride	*36*	*24*	*33*	*18*	*100*	*24*	100	NR	NR
Naphthalene	*36*	*24*	*33*	*18*	*100*	*24*	4000	660	NR
n-Propylbenzene	*36*	*24*	*33*	*18*	*100*	*24*	NR	NR	NR
Styrene	*36*	*24*	*33*	*18*	*100*	*24*	3000	NR	NR
Tetrahydrofuran	*36*	*24*	*33*	*18*	*100*	*24*	NR	NR	NR
trans-1,4-Dichloro-2-butene	*36*	*24*	*33*	*18*	*100*	*24*	NR	NR	NR
1,1,2-Trichloro-1,2,2-trifluoroethane	*36*	*24*	*33*	*18*	*100*	*24*	NR	NR	NR
1,2,3-Trichloropropane	*36*	*24*	*33*	*18*	*100*	*24*	NR	NR	NR
1,1,1,2-Tetrachloroethane	*36*	*24*	*33*	*18*	*100*	*24*	100	25	NR
1,1,2,2-Tetrachloroethane	*36*	*24*	*33*	*18*	*100*	*24*	5	5	NR
Tetrachloroethene (PCE)	*36*	*24*	*33*	*18*	*100*	*24*	1000	370	NR
Toluene	*36*	*24*	*33*	*85*	*100*	*24*	30000	1300	NR
1,2,3-Trichlorobenzene	*36*	*24*	*33*	*18*	*100*	*24*	NR	NR	NR

Table X. (Continued)

Analyte	SC1-Comp	SC2-Comp	SC3-Comp	MR-Comp	MB-Comp	WC-Comp	MCP[1]	BUD[2]	Lined Landfill[3]
1,2,4-Trichlorobenzene	*36*	*24*	*33*	*18*	*100*	*24*	2000	660	NR
1,1,1-Trichloroethane	*36*	*24*	*33*	*18*	*100*	*24*	30000	1900	NR
1,1,2-Trichloroethane	*36*	*24*	*33*	*18*	*100*	*24*	100	5	NR
Trichloroethene (TCE)	*36*	*24*	*33*	*18*	*100*	*24*	300	110	NR
Trichlorofluoromethane	*36*	*24*	*33*	*18*	*100*	*24*	NR	NR	NR
1,2,4-Trimethylbenzene	*36*	*24*	*33*	*18*	*100*	*24*	NR	NR	NR
1,3,5-Trimethylbenzene	*36*	*24*	*33*	*18*	*100*	*24*	NR	NR	NR
Vinyl chloride	*36*	*24*	*33*	*18*	*100*	*24*	600	280	NR
o-Xylene	*36*	*24*	*33*	*18*	*100*	*24*	NR	420	NR
m,p-Xylenes	*73*	*47*	*67*	*36*	*210*	*48*	NR	420	NR
PCB's by 8082 (ug/kg):									
Aroclor 1016	*110*	*59*	*100*	*57*	*150*	*120*	2000*	44	NR
Aroclor 1221	*110*	*59*	*100*	*57*	*150*	*120*	2000*	44	NR
Aroclor 1232	*110*	*59*	*100*	*57*	*150*	*120*	2000*	44	NR
Aroclor 1242	*110*	*59*	*100*	*57*	*150*	*120*	2000*	44	NR
Aroclor 1248	*110*	*59*	*100*	*57*	*150*	*120*	2000*	44	NR
Aroclor 1254	*110*	*59*	*100*	480	*150*	*120*	2000*	44	NR
Aroclor 1260	*110*	*59*	*100*	*57*	*150*	*120*	2000*	44	NR
Pesticides by 8081A (ug/kg):									
Aldrin	*22*	*12*	*20*	*23*	*31*	*30*	40	22	NR
alpha-BHC	*22*	*12*	*20*	*23*	*31*	*30*	NR	NR	NR
beta-BHC	*22*	*12*	*20*	*23*	*31*	*30*	NR	NR	NR
delta-BHC	*22*	*12*	*20*	*23*	*31*	*30*	NR	NR	NR
gamma-BHC (Lindane)	*22*	*12*	*20*	*23*	*31*	*30*	NR	NR	NR
alpha-Chlordane	*22*	*12*	*20*	*23*	*31*	*30*	700	NR	NR
gamma-Chlordane	*22*	*12*	*20*	*23*	*31*	*30*	700	NR	NR
4,4'-DDD	*22*	*12*	*20*	*23*	*31*	*30*	4000	1800	NR
4,4'-DDE	*22*	*12*	*20*	*23*	*31*	*30*	3000	1300	NR
4,4'-DDT	*22*	*12*	*20*	*23*	*31*	*30*	3000	1300	NR
Dieldrin	*22*	*12*	*20*	*23*	*31*	*30*	50	23	NR
Endosulfan II	*22*	*12*	*20*	*23*	*31*	*30*	500	360	NR
Endrin aldehyde	*22*	*12*	*20*	*23*	*31*	*30*	NR	NR	NR
Endosulfan I	*22*	*12*	*20*	*23*	*31*	*30*	500	360	NR
Endosulfan sulfate	*22*	*12*	*20*	*23*	*31*	*30*	NR	NR	NR
Endrin	*22*	*12*	*20*	*23*	*31*	*30*	8000	3900	NR
Endrin ketone	*22*	*12*	*20*	*23*	*31*	*30*	NR	NR	NR
Heptachlor	*22*	*12*	*20*	*23*	*31*	*30*	200	96	NR
Heptachlor epoxide	*22*	*12*	*20*	*23*	*31*	*30*	90	56	NR
Methoxychlor	*22*	*12*	*20*	*23*	*31*	*30*	200000	76000	NR
Toxaphene	*1100*	*590*	*1000*	*1100*	*1500*	*1500*	NR	NR	NR
Chlordane	*110*	*59*	*100*	*110*	*150*	NR	700	700	NR
Polynuclear Aromatic HC (mg/kg):									
2-Methylnaphthalene	*1.67*	*1.12*	*1.94*	*0.97*	*2.57*	*1.89*	4000	660	NR
Acenaphthene	*1.67*	*1.12*	*1.94*	*0.97*	*2.57*	*1.89*	4000	3900	NR
Acenaphthylene	*1.67*	*1.12*	*1.94*	*0.97*	*2.57*	*1.89*	1000	1100	NR
Anthracene	*1.67*	*1.12*	*1.94*	*0.97*	*2.57*	*1.89*	1000000	1000000	NR
Benz(a)anthracene	*1.67*	*1.12*	*1.94*	*0.97*	*2.57*	*1.89*	7000	3700	NR
Benzo(a)pyrene	*1.67*	*1.12*	*1.94*	*0.97*	*2.57*	*1.89*	2000	660	NR
Benzo(b)fluoranthene	*1.67*	*1.12*	*1.94*	1.11	*2.57*	*1.89*	7000	3700	NR
Benzo(ghi)perylene	*1.67*	*1.12*	*1.94*	*0.97*	*2.57*	*1.89*	1000000	1000000	NR
Benzo(k)fluoranthene	*1.67*	*1.12*	*1.94*	*0.97*	*2.57*	*1.89*	70000	3700	NR
Chrysene	*1.67*	*1.12*	*1.94*	1.23	*2.57*	*1.89*	70000	370000	NR
Dibenz(a,h)anthracene	*1.67*	*1.12*	*1.94*	*0.97*	*2.57*	*1.89*	700	660	NR
Fluoranthene	*1.67*	*1.12*	*1.94*	1.89	*2.57*	*1.89*	1000000	1000000	NR
Fluorene	*1.67*	*1.12*	*1.94*	*0.97*	*2.57*	*1.89*	1000000	1000000	NR
Indeno(1,2,3-cd)pyrene	*1.67*	*1.12*	*1.94*	*0.97*	*2.57*	*1.89*	7000	3700	NR
Naphthalene	*1.67*	*1.12*	*1.94*	0.97	*2.57*	*1.89*	4000	660	NR
Phenanthrene	*1.67*	*1.12*	*1.94*	*0.97*	*2.57*	*1.89*	10000	10000	NR
Pyrene	*1.67*	*1.12*	*1.94*	1.82	*2.57*	*1.89*	1000000	1000000	NR
MA EPH Ranges - mg/kg-dry									
C11-C22 Aromatics	*66.6*	*44.8*	*77.5*	64	*103*	112	1000	480	NR
C09-C18 Aliphatics	*66.6*	*44.8*	*77.5*	*38.8*	*103*	*75.6*	1000	780	NR
C19-C36 Aliphatics	*66.6*	*44.8*	*77.5*	46.9	*103*	*75.6*	3000	3000	NR
Total TPH							NR	NR	100
1-Chlorooctadecane (%REC)	64	86	66	71	67	52	NR	NR	NR
o-Terphenyl (%REC)	73	51	70	73	72	71	NR	NR	NR

Italicized values aligned right = analyte not detected; value reported is laboratory detection limit

NR: Not Reported

*Standard applies to total PCBs

Analyte Exceeds MCP S-1/G-1Standard

Analyte Exceeds BUD Standard

Detection Limit Exceeds MCP Standard

1: MADEP, 2007. Massachusetts Contingency Plan 310 CMR 40

2: MADEP, 2004. Draft Interim Guidance Document for Beneficial Use Determination Regulations 310 CMR 19.060

3: MADEP, 1997. Reuse and Disposal of Contaminated Soil at Massachusetts Landfills Department of Environmental Protection Policy # COMM-97-001

Appendix E

Management Timeline and Cost Summary Tables

Management Timeline Summary Table

Management Technique	Where	Phase 1	Phase 2	Phase 3	Phase 4	Phase 5	Ongoing
Hydroraking	• Western cove of Main Basin (method has had previous short-term success) • West Cove	• Working Group to evaluate value for creating boating channels or other small-scale purpose	• Implement hydroraking, if needed	• Same as Phase 2	• Same as Phase 2	• Same as Phase 2	• Evaluate use for control of dense water lily beds and implement, if needed
Drawdown	• Main Basin and South Cove are best candidates • West Cove would need culvert rebuild or pumping to draw down	• Investigate ability to draw down pond and hydraulic feasibility • Investigate permitting issues • Obtain funding for permitting and design • Hire consultant(s) to conduct studies	• Drawdown Feasibility Study • Drawdown Operations Plan • Permitting • Conduct winter drawdown if permitting completed in time • Monitoring	• Conduct winter drawdown if not conducted in Phase 2 or as necessary and recommended • Monitoring	• Conduct winter drawdown as necessary and recommended • Monitoring	• Same as Phase 4	• Drawdown to be repeated as needed and as permitting allows • Annual monitoring to include recommendations on schedule for next drawdown
Dredging	• South Cove is best candidate – first priority will be northern portion, second priority will be southern portion • West Cove, and western cove of Main Basin also possible but lower priority	• Develop scope for proposed dredge program in South Cove • Obtain funding for permitting and design • Hire consultant(s) for sediment testing, engineering design and permitting	• Conduct sediment testing South Cove • Develop engineering design for submittal to permitting authorities • File for permits: ENF (MEPA), 401 Water Quality Certificate (DEP), Order of Conditions (Con Comm), Chapter 91 (DEP), Section 404 Permit (Army Corps)	• Complete permitting efforts • Obtain funding for Phase I of dredging program • If permitting complete and funding available, begin Phase I of dredge program (northernmost section of South Cove)	• Continue additional phases of dredge program, if needed and funding allows	• Continue additional phases of dredge program, if needed and funding allows	• Dredge program should be effective for decades • Update bathymetry map in South Cove every five years or as necessary (e.g., after extreme weather events)
Monitoring & Reporting	• Target management areas • Pondwide	• Monitor effectiveness on control of aquatic vegetation • Monitor impacts to non-target resources • Provide recommendations for future years	• Same as Phase 1	• Same as Phase 1	• Same as Phase 1	• Same as Phase 1	• Annual program to monitor and assess effectiveness and prioritize ongoing management
Chemical Control (Partial Lake or Spot Herbicide Treatment)	• Flumioxazin – any basin • Fluridone – easiest in West Cove but possible elsewhere	• Working Group to further investigate herbicides, especially with regard to herring conflicts	• Amend existing OOC or file new NOI, as needed, for flumioxazin treatment • Fluridone treatment for West Cove or elsewhere, as needed • Flumioxazin spot treatment or partial lake treatment, as needed • Monitoring	• Flumioxazin spot treatment or partial lake treatment as needed • Monitoring	• Same as Phase 3	• Same as Phase 3	• Chemical spot treatment or partial lake treatment to be repeated as needed and as permitting and funding allow • Ongoing control would be primarily or entirely achieved through a combination of other methods • Annual monitoring to include recommendations on schedule and targeted treatment areas
Upgrade/Maintain Town Infrastructure (Stormwater)	• Old Swamp River SNUP • West Cove stormwater drains • Watershed-wide	• Review stormwater BMP maintenance practices and schedules; adjust as needed	• Review need for stormwater improvements at Cynthia Circle outfall (West Cove) • Review SNUP effectiveness and optimization (Old Swamp River)	• Review need for changes to SNUP	• Pursue changes to SNUP and Cynthia Circle outfall as needed (ongoing)	• Identify opportunities for stormwater retrofits	• Continue to reduce sediment and nutrient inputs from town facilities and private properties
Novel Approaches (e.g., Limno-barriers)	• Typically associated with herbicide treatments	• No action identified	• Review need for limno-barriers (for pilot study or to isolate treated coves) • Purchase and deploy necessary length of limno-barriers, as desired and permitted • Remove and store limno-barriers when not in active use	• Review need to deploy limno-barriers • Install limno-barriers, as needed • Remove and store limno-barriers when not in active use	• Same as Phase 3	• Same as Phase 3	• Evaluate use for control of nuisance plants and implement, as needed

Management Timeline Summary Table

Management Technique	Where	Phase 1	Phase 2	Phase 3	Phase 4	Phase 5	Ongoing
Diver-Assisted Suction Harvesting (DASH) or Hand Harvesting	• DASH in deeper beds of any basin • DASH could be used to clear boating channels in western cove of Main Basin or West Cove • Hand harvesting along immediate shoreline of residences/public access areas	• Working Group and WPA to identify suitable areas, if any • Obtain funding for permitting and implementation	• Working Group and WPA to identify suitable areas, if any • Permitting • Conduct harvesting in target areas, if applicable	• Working Group and WPA to identify suitable areas, if any • Conduct harvesting in target areas, if applicable	• Same as Phase 3	• Same as Phase 3	• Annual monitoring to include recommendations on targeted treatment areas
Resident Waterfowl Control (e.g., Goose Barriers)	• Passive techniques at Middle Street access and private shorelines • Active techniques, if used, would take place where birds or nests are present	• Public education program to include information in kiosk, website, mailings, WPA	• Same as Phase 1	• Working Group to evaluate techniques, feasibility, cost for fence/barriers on town land • Permitting, if necessary	• Implementation on town land as agreed to	• Same as Phase 4	• Continue public education program and implementation on town land • Annually re-assess maintenance needs of structures and plantings • Annual monitoring of nuisance waterfowl populations
Benthic Barriers	• Western cove of Main Basin or West Cove to maintain boating channels or deeper waters near beaches • Private shorelines	• No action identified	• Same as Phase 1	• Working Group to evaluate techniques, applicability, feasibility, costs, schedule, etc. • Permitting	• Implementation as agreed to by Working Group (e.g., near public boat ramp or beach areas)	• Same as Phase 4	• Additional installations and maintenance/replacement of installations as needed
Biological Controls (for Purple Loosestrife only)	• Purple loosestrife growths at mouth of Old Swamp River (South Cove) and West Cove (if contiguous beds are present)	• Working Group to evaluate feasibility and cost • Purchase and release loosestrife beetles in key control areas	• Release loosestrife beetles in key control areas to reinforce population, as needed	• Same as Phase 2	• Same as Phase 2	• Same as Phase 2	• Annual monitoring to include recommended adjustments to this program
Aeration	• South Cove, if needed for algae control	• Working Group to evaluate continued operation of existing aerator	• No recommended action	• No action identified	• No action identified	• No action identified	• Review need for aeration at South Cove (if algae issues emerge)
Public Education & Involvement	• Watershed residents and visitors	• WPA, Working Group, Con Comm to prioritize program	• Identify, develop and disseminate educational materials • Conduct volunteer training as needed	• Same as Phase 2	• Same as Phase 2	• Same as Phase 2	• Ongoing program, working with WPA, Working Group, Con Comm, DPW, etc.
Boat Monitor or Weed Watchers Program	• Boat monitors at access locations • Weed Watchers pondwide	• Working Group to evaluate feasibility of sponsoring program and coordinate with WPA and appropriate town department(s)	• Coordinate with DCR Lakes and Ponds, WPA, and appropriate town department(s) to implement chosen program	• Same as Phase 2	• Same as Phase 2	• Same as Phase 2	• Review participation and impact of program on a regular basis and adjust, as necessary
Boating Rules (e.g., boating channels)	• Main Basin • Could be used in West Cove if conditions improved for boating	• Working Group to evaluate benefits and feasibility of new boating rules in Whitman's Pond	• Develop and implement new boating rules, if feasible	• Implement boating rules, if feasible	• Same as Phase 3	• Same as Phase 3	• Review boating rules on a regular basis and update, as necessary
No-action Alternative	• NA	• No action identified	• Same as Phase 1	• Same as Phase 1	• Same as Phase 1	• Same as Phase 1	• Same as Phase 1

Cost Summary Table

Management Technique	Estimated Costs				
	Studies	**Design/Permitting***	**Implementation/Activity**	**Ongoing Costs**	**Monitoring****
Hydroraking	$2,500 for independent pre-treatment mapping update	*Already permitted under existing OOC*	$6,000 – more than $12,000/acre	Repeat every 3-5 yrs	$4,500-$8,000 for annual monitoring
Drawdown	$8,000 for Drawdown Operations Plan	$6,000 to $8,000	No cost, unless alteration of discharge structures required	Ongoing operational costs dependent on need for pumping, if any	$7,000 for annual monitoring
Dredging: South Cove <u>Northern portion South Cove only</u> (75,000-100,000 cy) - Dry dredge - Hydraulic dredge <u>Entire South Cove</u> (275,000 cy) - Dry or hydraulic dredge	 Included in implementation costs Included in implementation costs Included in implementation costs	 $100,000 - $120,000 (depends on number of sediment samples required) $175,000 - $200,000 (cost difference associated with sediment sampling)	 $1.5 Million - $2.5 Million $1.9 Million - $4.5 Million (varies due to dewatering and disposal methods) $4.5 Million - $6 Million (disposal costs likely to increase this figure)	 No cost	 No technique-specific monitoring usually necessary
Dredging: West Cove (150,000 cy) - Dry or hydraulic dredge	Included in implementation costs	$125,000 - $150,000	$3.0 Million - $5.0 Million	No cost	No technique-specific monitoring usually necessary
Dredging: Western Portion of Main Basin (200,000 cy) - Dry or hydraulic dredge	Included in implementation costs	$150,000 - 175,000	$4.0 Million - $5.5 Million	No cost	No technique-specific monitoring usually necessary
Annual Monitoring (plants, other biology, basic water chemistry)	No cost	No permit required	$2,500 for initial QAPP (if needed)	$5,000 - $8,000/year	See ongoing costs

Cost Summary Table

Management Technique	Estimated Costs				
	Studies	**Design/Permitting***	**Implementation/Activity**	**Ongoing Costs**	**Monitoring****
Chemical Control (Partial Lake or Spot Herbicide Treatment)	$2,500 for initial pre-treatment mapping update	*Sonar already permitted under existing OOC* Re-permitting for Clipper $5,000 as standalone NOI to Con Comm	$15,000 - $35,000/basin for partial lake treatment $8,000 - $30,000 for partial treatment (key recreation areas) $3,000+ for spot treatments	Repeat treatments as needed Typically $3,000 - $10,000+ annually, depending on areas treated and formulation	$4,500-$8,000 for annual monitoring
Upgrade/Maintain Town Infrastructure (Stormwater)	Cost varies widely May conduct study to prioritize BMP locations by potential to remove contaminants. Cost for such a study would be $5,000 to over $25,000 depending on scope.	Design and permitting of BMPs vary widely in cost and effectiveness	Varies widely depending on design of BMPs and site constraints	Quarterly to annual maintenance costs typically associated with most BMPs	Monitoring usually incorporated into Operation and Maintenance Plan or required under the Town's MS4 NPDES permit
Novel Approaches (e.g., Limno-barriers)	No cost	Include in NOI prepared for herbicide use	$10-$15/linear foot for material $2,000 for a one-day installation	Negligible maintenance costs. Installation and removal could be done by contractor, Town, or volunteers	See chemical control
Diver Assisted Suction Harvesting (DASH) or Hand Harvesting	$2,500 for independent pre-treatment mapping update	$5,000 as standalone NOI to Con Comm	>$2,000 - $5,000/acre	Repeat as needed	$5,000-$8,000 for annual monitoring
Resident Waterfowl Control (e.g., Goose Barriers)	Included in permitting costs	Varies from no cost to $5,000 for NOI to Con Comm (depends on technique)	$4 – 15/linear foot (applies to fencing only)	Varies widely by technique	$2,500-$5,000 for annual monitoring
Benthic Barriers	No cost	If over large area or for general control, $5,000 for NOI to Con Comm	$2/square foot	Repeat as material breaks down.	$2,500-$5,000 for annual monitoring
Biological Controls (for Purple Loosestrife only)	$2,500 for baseline mapping	No cost – beetles must be obtained from permit holder	$275 - $300/1,000 beetles	Repeat as needed to ensure control	$2,500-$5,000 for annual monitoring

Cost Summary Table

Management Technique	Estimated Costs				
	Studies	**Design/Permitting***	**Implementation/Activity**	**Ongoing Costs**	**Monitoring****
Aeration	No studies required for small scale aeration (around docks) Otherwise, $2,500-$5,000	Varies from negligible for small scale aeration to $5,000 as standalone NOI to Con Comm	Costs vary widely for aeration systems and can exceed $20,000 per unit.	Costs vary widely, depending on source of aeration, energy required, and whether units need to be removed	No technique-specific monitoring usually required
Public Education and Involvement	NA	Permit not usually required but varies by activity	Costs vary widely for educational materials and training	Costs vary widely for educational materials and training	No technique-specific monitoring usually necessary
Boat Monitor or Weed Watchers Program	No cost	No permit required	No mandatory costs	No mandatory costs	Incorporate into annual monitoring program
Boating Rules (e.g., boating channels)	No cost	No permit required	Costs for signage/public education, as needed	Enforcement cost (applicable to mandatory rules only)	No technique-specific monitoring usually necessary
No-action Alternative	No monetary cost	No monetary cost	No monetary cost	No monetary cost	No monetary cost

*Significant cost savings possible if local permitting completed simultaneously for multiple management actions

**Costs are not necessarily additive. Rather, they reflect costs to implement a monitoring program for a standalone management technique. Some monitoring costs may be shared across management techniques. Final cost depends on scope of the monitoring program, including any permit-conditioned elements.

Appendix F

Summary of Public Comments Received

WHITMAN'S POND VEGETATION MANAGEMENT PLAN
PUBLIC MEETING – JUN 10, 2013

Notes compiled by ME Schloss, 7/10/13, revised 7/15/13

Public Meeting: Recap of Public Comments Received

- Approximately 30 people attend, in addition to Working Group
- Comments focused on:
    - Lack of action: why is it taking so long to do something?
    - Can't use waterfront property
    - Paying taxes for waterfront
    - Don't want herbicides in South Cove or other areas of the pond
    - Can residents manage water areas in front of their own properties?
    - Can we train resident volunteers for hand pulling, weed monitoring, etc?
    - West Cove filling in rapidly by mobile home and cemetery area.
    - Hydroraking is best option
    - Concern about impacts of drawdown on overall ecology of the Back River.
    - Concern re: biological control with loosestrife beetles: what happens when they finish eating all the loosestrife?
    - Dredging is best option for West Cove and other areas. Is a long-term solution. Chemicals are not. Will increasing depth of water help with water supply capacity?
    - Need to address sedimentation coming into the pond from stormwater runoff.
    - How can we empower homeowners and residents? Can we get a blanket permit to allow them to manage activities in front of their properties?
    - Agreement that the problem requires a multi-faceted approach
    - Are unsewered properties around the pond significantly contributing to the problem? Should we be looking at that?
    - Should we be considering aeration to oxygenate the "dead zone"?
    - Why is the state stocking the pond with fish if it is not suitable fish habitat?
    - What is the impact of these approaches on the herring? This question must be addressed prior to permitting, as perceived impacts derailed the last proposal.
    - Concerned we are backing into more expensive alternatives (in the move away from chemical treatment).
    - Back River Watershed Association is concerned about adverse impacts of chemical treatment. Hoping for long-term approach, and agree may need to be multiple approaches taken. BRWA would like to work with Whitman's Pond Association on the issues – we all need to work together.
    - Is there anything residents can do during summer drought periods to remove plants when shoreline is exposed?

- Should be doing better outreach and education for boaters.
- Boat monitors are needed to inspect boats and trailers. People aren't doing it – can take an hour to clean off the weeds! Also to discourage boaters that come in from salt water just to discharge their bilge water into Whitman's Pond so they don't have to clean it out at home. Has seen larger, sea-going boats just come into the pond to drive around once and leave. Sure this is what they are doing.
- Can town install a trash barrel at the boat ramp (near the private house)?

Public comments received after meeting:

From Maria Shapiro of Greenvale Ave and Rob Stevens of West Lake Drive

- Concern about West Cove filling in. Lots worse than it used to be. Paying waterfront taxes but is turning into a swamp.
- Why no action in so much time?
- Can residents help to hand pull the weeds?
- Need to publicize meeting summary and decision of the Working Group.
- Infrastructure improvements needed (stormwater)
- Stormwater inputs is huge issue in West Cove
- Better to permit all dredging activities up front, for all 3 basins (see p. 53 of report).
- If dredging, can dredging sediments be used to create a new shoreline? Will this provide additional habitat? Additional public amenity? Reduce cost?
- Need to sell the project to the public.
- Working Group should look at public access – what exists, are more opportunities needed?
- Can we include activities that residents can do in the town's permit?
- How bad are failing septic systems around the pond? Can town help with low-interest loan program?
- Can we use the town's Capital Improvement Program to do some of this work?
- Can we get grants for stormwater improvements?
- Can the town increase its CPC assessment for a short period of time only (e.g., 5 years) to help pay for some of these improvements?
- Could the town float a bond and have the CPC pay the debt service?
- Can we open the pond for swimming again?
- Can we at least dredge a channel through West Cove?
- Need to reach out to homeowners for general education: do's and dont's
- Citizen training, weed watchers, volunteer opportunities (e.g., weed pulling once a month in the morning….)
- Reach out to Weymouth HS science dept
- Need short-term strategy to show action!

Appendix G

Glossary of Terms

APPENDIX G - GLOSSARY OF LIMNOLOGICAL TERMS

Abiotic: A term that refers to the nonliving components of an ecosystem (e.g., sunlight, physical and chemical characteristics).

Algae: Typically microscopic plants that may occur as single-celled organisms, colonies or filaments.

Anoxic: Greatly deficient in oxygen.

Aquifer: A water-bearing layer of rock (including gravel and sand) that will yield water in usable quantity to a well or spring.

Aquatic plants: A term used to describe a broad group of plants typically found growing in water bodies. The term may generally refer to both algae and macrophytes, but is commonly used synonymously with the term macrophyte.

Bacteria: Typically single celled microorganisms that have no chlorophyll, multiply by simple division, and occur in various forms. Some bacteria may cause disease, but many do not and are necessary for fermentation, nitrogen fixation, and decomposition of organic matter.

Bathymetric Map: A map illustrating the bottom contours (topography) and depth of a lake or pond.

Best Management Practices: Any of a number of practices or treatment devices that reduce pollution in runoff via runoff treatment or source control.

Biomass: A term that refers to the weight of biological matter. Standing crop is the amount of biomass (e.g., fish or algae) in a body of water at a given time. Biomass is often measured in grams per square meter of surface.

Biovolume: Analogous to biomass but expressed in terms of volume rather than mass.

Biota: All living organisms in a given area.

Chlorophyll a: A pigment used by higher plants and certain algae for photosynthesis. Measuring the level of this pigment in surface water is one way of describing the productivity of a pond and determining its trophic state (see Eutrophic).

Cultural Eutrophication: The acceleration of the natural eutrophication process caused by human activities, occurring over decades as opposed to thousands of years.

Ecosystem: An interactive community of living organisms, together with the physical and chemical environment they inhabit.

Endangered/Threatened Species: An animal or plant species that is in danger of extinction and is recognized and protected by state or federal agencies.

Erosion: A process of breakdown and movement of land surface that is often intensified by human disturbances.

Eutrophic: A trophic state (degree of eutrophication) in which a lake or pond is nutrient rich and sustains high levels of biological productivity. Dense macrophyte growth, fast sediment accumulation, frequent algae blooms, poor water transparency and periodic oxygen depletion in the hypolimnion are common characteristics of eutrophic lakes and ponds.

Eutrophication: The process, or set of processes, driven by nutrient, organic matter, and sediment addition to a pond that leads to increased biological production and decreased volume. The process occurs naturally in all lakes and ponds over thousands of years.

Exotic Species: Species of plants or animals that occur outside of their normal, indigenous ranges and environments. Populations of exotic species may expand rapidly and displace native populations if natural

predators, herbivores, or parasites are absent or if conditions are more favorable for the growth of the exotic species than for native species.

Filamentous: A term used to refer to a type of algae that forms long filaments composed of individual cells.

Groundwater: Water found beneath the soil surface and saturating the layer at which it is located.

Habitat: The natural dwelling place of an animal or plant; the type of environment where a particular species is likely to be found.

Herbicide: Any of a class of chemical compounds that produce mortality in plants when applied in sufficient concentrations.

Hypoxic: Lacking sufficient dissolved oxygen to support all but the most tolerant species.

Infiltration Structures: Any of a number of structures used to treat runoff quality or control runoff quantity by infiltrating runoff into the ground. Includes infiltration trenches, dry wells, infiltration basins, and leaching catch basins.

Invasive: Spreading aggressively from the original site of planting.

Isopach Map: A map illustrating the thickness of sediments within a lake or pond.

Limnology: The study of lakes.

Littoral Zone: The shallow, highly productive area along the shoreline of a lake or pond where rooted aquatic plants grow.

Macroinvertebrates: Aquatic insects, worms, clams, snails and other animals visible without aid of a microscope. They supply a major portion of fish diets and are important consumers of detritus and algae.

Macrophytes: Macroscopic vascular plants present in the littoral zone of lakes and ponds.

Morphometry: A term that refers to the depth contours and dimensions (topographic features) of a lake or pond.

Nonpoint Source: A source of pollutants to the environment that does not come from a confined, definable source such as a pipe. Common examples of nonpoint source pollution include urban runoff, septic system leachate, and runoff from agricultural fields.

Nutrient Limitation: The limitation of growth imposed by the depletion of an essential nutrient.

Nutrients: Elements or chemicals required to sustain life, including carbon, oxygen, nitrogen and phosphorus.

pH: An index derived from the inverse log of the hydrogen ion concentration that ranges from zero to 14 indicating the relative acidity or alkalinity of a liquid.

Photosynthesis: The process by which plants use chlorophyll to convert carbon dioxide, water and sunlight to oxygen and cellular products (carbohydrates).

Phytoplankton: Algae that float or are freely suspended in the water.

Pollutants: Elements and compounds occurring naturally or man-made introduced into the environment at levels in excess of the concentration of chemicals naturally occurring.

Secchi disk: A black and white or all white 20 cm disk attached to a cord used to measure water transparency. The disk is lowered into the water until it is no longer visible (Secchi depth). Secchi depth is generally proportional to the depth of light penetration sufficient to sustain algae growth.

Sediment: Topsoil, sand, and minerals washed from the land into water, usually after rain or snowmelt.

Septic system: An individual wastewater treatment system that includes a septic tank for removing solids, and a leachfield for discharging the clarified wastewater to the ground.

Siltation: The process in which inorganic silt settles and accumulates at the bottom of a lake or pond.

Stormwater Runoff: Runoff generated as a result of precipitation or snowmelt.

Temperature Profile: A series of temperature measurements collected at incremental water depths from surface to bottom at a given location.

Thermal Stratification: The process by which a lake or pond forms several distinct thermal layers. The layers include a warmer well-mixed upper layer (epilimnion), a cooler, poorly mixed layer at the bottom (hypolimnion), and a middle layer (metalimnion) that separates the two.

Thermocline: A term that refers to the plane of greatest temperature change within the metalimnion. Often used interchangeably with metalimnion.

TKN: Total Kjeldahl nitrogen, essentially the sum of ammonia nitrogen and organic forms of nitrogen.

TSS: Total suspended solids, a direct measure of all suspended solid materials in the water.

Turbidity: A measure of the light scattering properties of water; often used more generally to describe water clarity or the relative presence or absence of suspended materials in the water.

Vegetated Buffer: An undisturbed vegetated land area that separates an area of human activity from the adjacent water body; can be effective in reducing runoff velocities and volumes and the removal of sediment and pollutant from runoff.

Water Column: Water in a lake or pond between the interface with the atmosphere at the surface and the interface with the sediment at the bottom.

Water Quality: A term used to reference the general chemical and physical properties of water relative to the requirements of living organisms that depend upon that water.

Watershed: The surrounding land area that drains into a water body via surface runoff or groundwater recharge and discharge.

Zooplankton: Microscopic animals that float or are freely suspended in the water.